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The undersigned hereby certify that they have read and recommend to the Committee on Graduate Studies for acceptance, a dissertation on "Some Physical Properties of Alberta Coal" submitted by John Ernest Oberholtzer, B. Sc. in partial fulfilment of the requirements for the degree of Master of Science.

Professor

Professor

Professor

S O M E P H Y S I C A L P R O P E R T I E S O F
A L B E R T A C O A L S

- by -

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Research Council of Alberta

University of Alberta

A T H E S I S

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in partial fulfilment of the require-
ments for the degree of Master of Science.


Edmonton, Alberta.

April 1941.

P R E F A C E A N D A C K N O W L E D G E M E N T S

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C H A P T E R I.

GENERAL INTRODUCTION.

The investigations here described all fall under the general title of the Physical Properties of Alberta Coals. Much work related to these properties of coal in general has been done in various parts of the world, while particular studies along these lines relating to Alberta coals have been in progress for several years in the laboratories of the Research Council of Alberta. Still the field is far from exhausted, and much work is yet to be done.

Several aspects of the topic have been considered, and the work described has been divided into three main sections.

The first investigation dealt with the moisture holding properties of Alberta coals. This was a continuation of work previously done in these laboratories and entailed:

- (a) The determination and redetermination of the vapor pressures of the saturated solutions of various salts at a definite temperature
- (b) The determination of the moisture holding properties of various samples of coal with the use of these salt solutions.

This problem was not included in the original plan of research, but was necessitated by the request from the Dominion Government Fuel Testing laboratories in Ottawa for a cooperative investigation.

The second investigation dealt with the wetting of coal dust

by water, and involved the testing of several wetting agents now on the market. This work was carried out with a view toward its possible usefulness in coal mines for the prevention of dust explosions, and because the wetting of coal was required as part of the technique of later work.

The third, and possibly the most important investigation, was concerned with very finely ground coal, sometimes called "u coal". The problems entailed here include:

- (a) The development of a method for evaluating the fineness of grinding.
- (b) The development of a method for determining the specific gravity of coal; this involved the use of the wetting agents mentioned above.
- (c) The use of a new ball mill for comparison of fine grinding of coal in air and in vacuo.

Earlier investigations in this laboratory on the oxidation of coal were temporarily suspended because it was found necessary to use finer grinding of coal than was then possible, and to have a method for the determination of surface exposed. The work here described will now permit the continuation of the oxidation studies.

It can be seen that every stage of this investigation is closely related to problems of importance to the coal industry of the province, and this relationship was kept in mind throughout.

C H A P T E R I I

CAPACITY MOISTURE OF COAL

1. Introduction.

The classification of coal now employed in North America, both by scientific workers and in commerce, involves, for the lower rank coals, their moisture holding character. Specifications for this classification are given by the American Society for Testing Materials under the heading "Classification of Coal by Rank" (1). These specifications, it might be noted, have been approved in Canada.

In the above specifications, coals containing less than 69 percent of fixed carbon on the dry, mineral-matter-free basis, are classified by their heat value on their "moist" mineral matter free basis. If the reference to moisture had stopped at this point, it might have resulted in coal from a dry section of a mine being given a higher classification than the same coal from a wet section of the mine. The specifications note, however, that "moist" refers to coal containing its natural bed moisture but not including visible water on the surface of the coal. They further state that "samples containing surface moisture shall be brought to a standard condition of moisture equilibrium at 30°C in a vacuum desiccator containing a saturated solution of K_2SO_4 (97 percent humidity) as suggested by Stansfield and Gilbert" (2). These specifications ensure that free surface moisture shall have no effect on the evaluation of the moisture content and only the "true moisture" shall be considered. (Stansfield and Gilbert later adopted the term "Capacity Moisture" as preferable to "True Moisture").

2. Early Work.

The procedure for the determination of capacity moisture of coal, embodied in the above A.S.T.M. specifications, is the result of a great deal of work, most of it done in the laboratories of the Research Council of Alberta. The findings of the earlier work have been published (3) but later developments have not been dealt with, so space is given here for a brief outline of previous work by E. Stansfield, K. C. Gilbert and others, leading up to the determination of the capacity moisture of coals.

In 1923 an air drying apparatus was constructed in these laboratories (3) for the dual purpose of preparing the sample for laboratory handling and of evaluating the moisture holding capacity of the coal. Here, crushed coal was dried in a stream of air of 60 percent humidity. This did not provide temperature control, and as air was used, the coal was liable to oxidation. Consequently a new apparatus was constructed early in 1931 which maintained a uniform temperature of 30°C and circulated, instead of air, natural gas of 60 percent humidity (4).

Other workers had been studying the relation between the moisture in coal and the vapor pressure of such moisture, and had made use of desiccators and constant-humidity solutions (5). Following along these lines, a vacuum desiccator method was developed in 1931 in these laboratories for the rapid routine determination of the capacity moisture of a coal as an evaluation of the bed moisture of the coal in the seam, unaffected by local conditions of free moisture in the mine.

The procedure (2) is outlined as follows. A fresh coal sample is crushed to pass a 14 mesh screen. Weighed portions of about 5 g. each are placed in aluminum capsules, and two capsules with covers removed are placed in each of a series of desiccators. Each of these desiccators has in its lower compartment a saturated solution of a salt plus crystals

of the salt itself. Such a system maintains a constant humidity at a given temperature. The relative humidities supplied by the desiccators employed, range from 12 percent to 97 percent. The desiccators are evacuated to avoid oxidation and to speed up the attainment of equilibrium of the system. They are then placed in a constant temperature chamber maintained at 30°C for 48 hours. The moisture still retained by each portion of coal, after coming to equilibrium with the humidity conditions of its desiccator, is then determined by drying in a vacuum oven for 3 hours at 105-110°C. The values thus found are plotted against the corresponding relative humidities, and a curve obtained, representing values up to a humidity of 97 percent. The curve is then extrapolated to cut the 100 percent humidity line and the point of intersection taken as the capacity moisture of the coal.

It was assumed by Stansfield and Gilbert "that the capacity moisture of the coal is the minimum amount of water in the fresh coal which can exert the same vapor pressure as free water at the same temperature". Hence the capacity moisture, determined as described, is the moisture value that should be used for classification purposes.

It was found after a large number of samples had been tested by this method, that the moisture retained by any coal in the desiccator containing K_2SO_4 rarely differed notably from 0.986 times the capacity moisture. 0.986 was the average value found with some 69 coals. Thus a much simplified method of determining capacity moisture could be used. Now the retained moisture of the coal sample in the K_2SO_4 desiccator only was determined, and the value divided by 0.986 to give the capacity moisture. This procedure was adopted as a routine test in these laboratories in 1934 and is still in use.

3. Current Investigation.

In 1939, however, J.H.H.Nicolls of the Fuels Division of the Canadian Bureau of Mines, challenged the accuracy of the factor $\frac{1}{0.986}$ (1.014) and suggested the use of the factor 1.03. Accordingly a further investigation along these lines was started in cooperation with the fuel testing laboratories in Ottawa and the Pittsburgh laboratories of the U. S. Bureau of Mines (6).

Scope. The investigation undertaken was divided into two main sections. First, the determination of the relative humidities obtained by the use of saturated solutions of various salts, and second, a determination of the capacity moistures of a number of coals by both the graphical and calculation methods described above.

In all, 15 salts were investigated, and the most suitable of these used in the determination of the capacity moistures of 54 coals.

Similar tests were made on most of the coals in the above laboratories of Ottawa and Pittsburgh.

A proximate analysis was carried out on each of the samples in addition to the moisture tests, in order to check the accuracy of sampling by comparison with proximate analyses made in Ottawa.

Determination of Relative Humidities. Stansfield and Gilbert list a number of salts with the corresponding relative humidities resulting from their saturated solutions as shown in table I. These were approximate values taken from the International Critical Tables, and extrapolated where necessary to 30°C.

Later, values for the actual solutions used were determined by Gilbert by the measured depression of the mercury column in a barometric tube with the introduction of water and of the saturated salt solutions.

His results are also shown in table I.

Gilbart lacked facilities for obtaining high accuracy, and his method was tedious; consequently redeterminations were made by a new method.

The new method finally adopted was that of air saturation. At first an attempt was made to measure the moisture taken up by measured volumes of air over water and over the solutions of salts tested, but this was found to be slow and the volume measurement inaccurate. Later a new procedure was tried and found satisfactory. This was essentially the method of Washburn and Heuse for the determination of vapor pressures (7) but with a simplification both of apparatus and procedure.

Air is drawn in a continuous stream through, first, a saturation system of water, second, a weighed drying train, third, a saturation system of the desired salt solution, and fourth, a second drying train. The whole is maintained at a constant temperature.

TABLE I
Relative Humidities of Saturated Salt Solutions at 30°C

Salt	Values as Percent	
	Int. Critical Tables	Determined (Gilbart)
LiCl.H ₂ O	12	11.0
KC ₂ H ₃ O ₂	20	19.5
MgCl ₂ .6H ₂ O	-	31.9
K ₂ CO ₃	44	-
Ca(NO ₃) ₂	-	46.8
NH ₄ NO ₃	60	59.7
NaCl	75	75.1
KCl	-	84.5
BaCl ₂ .2H ₂ O	88	89.9
K ₂ SO ₄	96	96.8
KClO ₃	-	97.7

The weight of moisture taken up by the air in each saturation system, and collected in the following drying train, depends upon the

humidity within the saturator and the volume of air passed through. As the volume of air passed through in any experiment is essentially the same in both the water and salt saturators, the relative humidity in the latter can be calculated from the determined moisture weights.

Apparatus. The apparatus consists of two saturation systems, two drying trains, and a suction bottle as shown in the flow diagram of figure I,1. Each saturation system consists of four tubes so arranged that the air flow passes from end to end of each of the four tubes connected in series. Each tube is constructed and charged as shown in figure I,2, though in the water saturation system the tubes contain only distilled water and glass wool.

Glass wool in the tubes increases the surface of liquid exposed to the air and decreases the time required for saturation.

Both sets of saturation tubes are mounted on a light wooden framework, which is rocked gently by an eccentric pulley arrangement run by an electric motor. This rocking ensures a saturated solution of the salt and exposure of freshly moistened surface at all times. The quantity of liquid and the violence of rocking are carefully regulated.

Each drying train consists of two U-tubes filled with dehydrite.

Figure II shows photographs of the apparatus used.

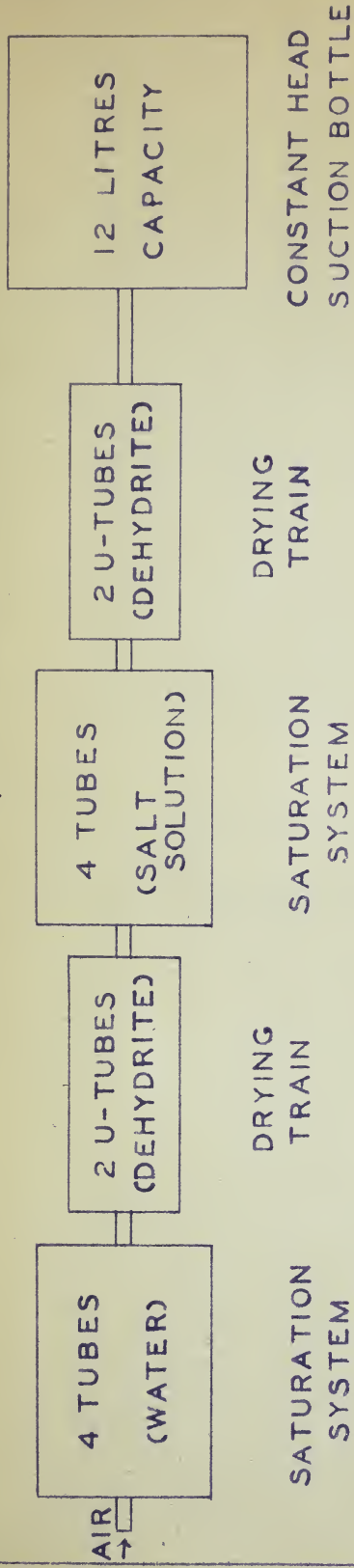
Procedure. The tubes of both saturation systems are charged, connected, and placed on the framework. The whole is then placed within a constant temperature chamber kept at 30°C. The rocking is started and continued for an hour without air flow to ensure a state of equilibrium throughout the system. The weighed drying trains and the suction bottle are then connected and air is drawn through the apparatus at the rate of approximately half a litre per hour for from 24 to 48 hours. A constant

Figure I.

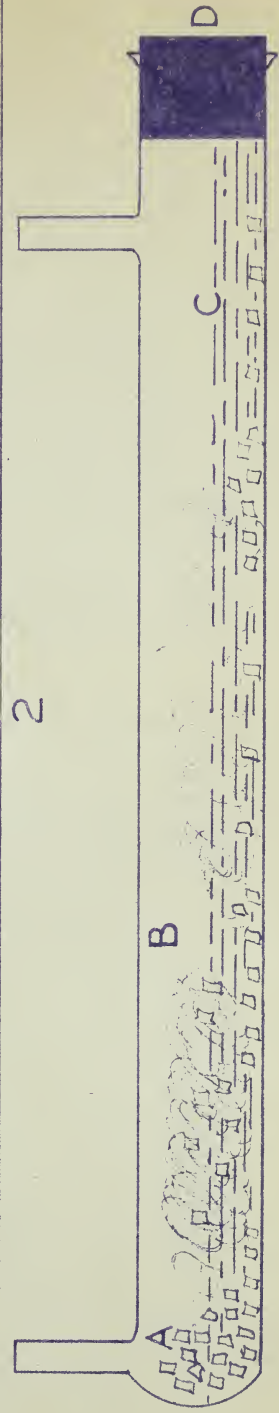
Apparatus for Determination of Relative Humidities
of Saturated Salt Solutions.

1. Flow diagram of apparatus.
2. Saturation tube - three-quarters of full size.

1



2



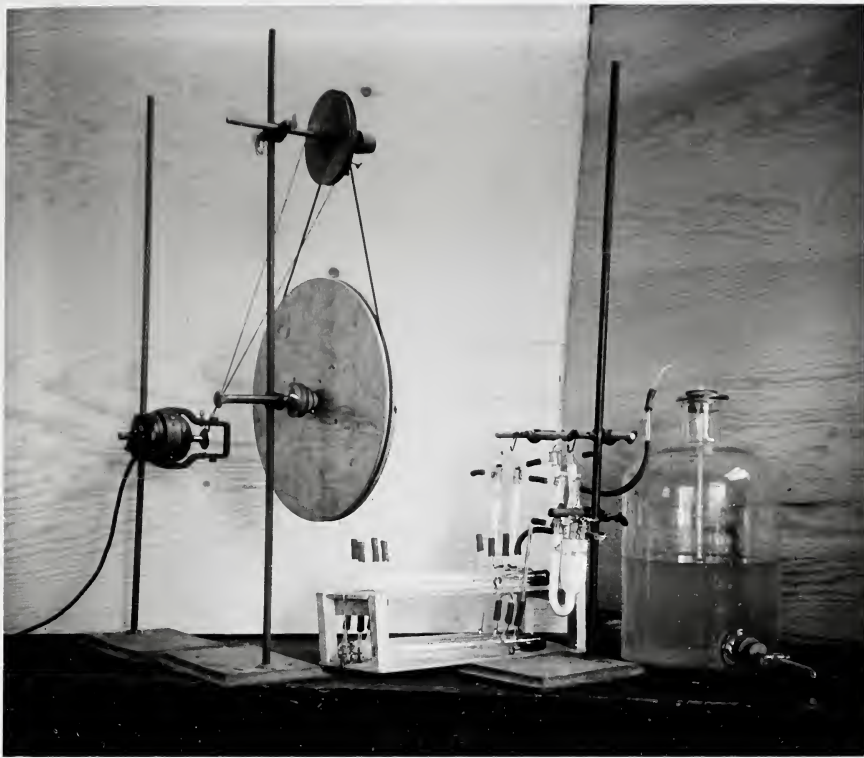
- A - SALT CRYSTALS
- B - GLASS WOOL
- C - SATURATED SALT SOLUTION
- D - RUBBER STOPPER

Figure II

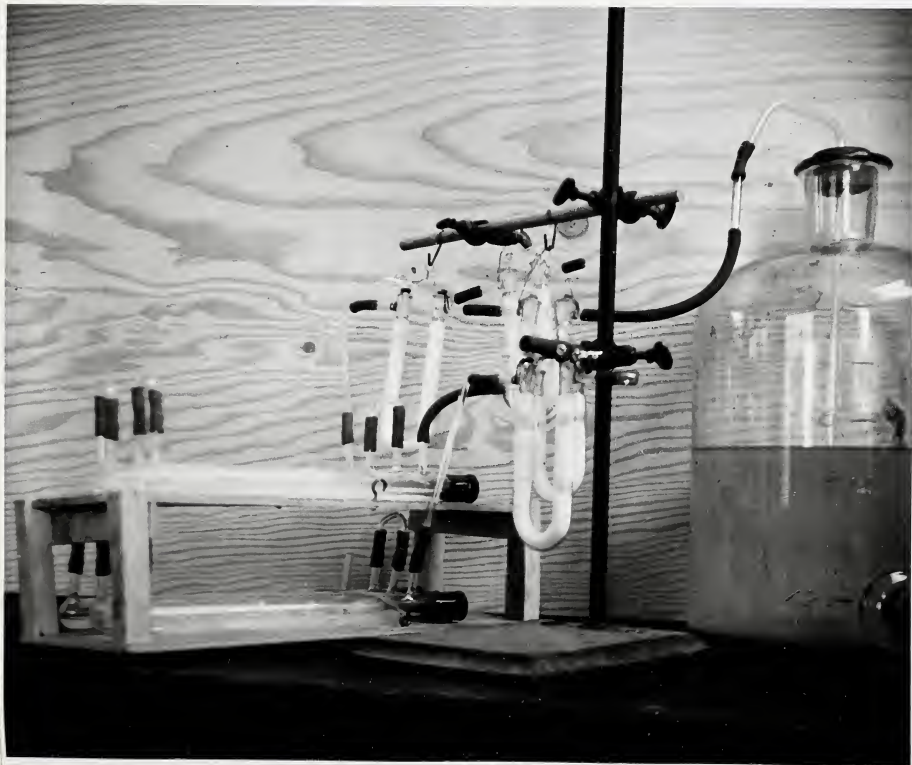
Photographs of Relative Humidity Apparatus.

Photograph 1 shows the complete apparatus with rocking arrangement.

Photograph 2 gives a closer view of the tubes on the framework and of the drying train.



Photograph 1.



Photograph 2.

head is maintained by the suction bottle, and the rate of flow is regulated by a pinch clamp. At the end of the run, the saturation systems are disconnected, and approximately half a litre of dry air is drawn through in order to collect any moisture that might remain in the side arms. Finally the drying trains are weighed.

Various precautions were taken to guard against experimental errors.

Temperature in the chamber was controlled to plus or minus 0.2°C . Before and after each run the system was tested for air tightness.

The saturated salt solutions were made with C.P. salts and distilled water, as it was not thought necessary to prepare purified samples of the salts. The same salts were used in the work for capacity moisture later described.

Salts giving a low relative humidity required the longer run of 48 hours to give a readily weighable increase in weight to the drying train, while for those of higher relative humidity 24 hours was sufficient.

Runs were first made with water in both saturation systems, with successive reductions in the rate of flow of air until concordant results indicated complete saturation of the air during its passage through the tubes. This concordance also indicated that the drop in pressure, and consequent increase in volume between the first and second saturation systems did not cause a measurable effect in the results. However, as a precaution, the two saturation systems were interchanged in position in alternate runs for every determination.

Calculations. The calculation of results is simple. If X is the

increase in weight of one drying train due to the moisture taken up from the salt solution, and Y is the corresponding increase in the weight of the other drying train due to the moisture taken up from the water, then the percent relative humidity of the salt solution system at 30°C is $\frac{X}{Y} \times 100$.

Example from notebook:

Salt in solution	Potassium sulphate
Length of run	24 hours
Approximate volume of air	12 litres
Weight of water taken from the salt solution tubes	= 0.2735 g.
Weight of water taken from the water tubes	= 0.2796 g.
Relative humidity = $\frac{0.2735}{0.2796} \times 100$	= 97.8%

Values Determined. Table II gives a list of the separate determinations and their averages. Results with several other salts which proved unsatisfactory for use have been excluded from the table. For example, potassium persulphate gave steadily decreasing values due to slow decomposition, and with barium nitrate, which has a relative humidity of over 99%, erratic results were obtained.

Discussion of Results. It will be noted that these values vary slightly from those determined by Gilbert. The results are reasonably consistent, with a maximum deviation from the average of approximately 0.5% and an average deviation of approximately 0.3% of the values.

As these values are used with moisture values whose allowable deviation by A.S.T.M. specifications (8) is approximately 3%, closer checking of results was not deemed necessary.

Determination of Capacity Moisture. Capacity moisture determinations were carried out on 54 coals, 11 from Saskatchewan, 11 from U.S.A. and 32 from Alberta. Table III lists etc. samples, indicating the source and classification of each. These were all lower rank coals for, as already stated, it is these coals that are classified on the

TABLE II
Relative Humidities of Saturated Salt Solutions at 30°C

Salt	Determined Value %	Average	Earlier Determination
LiCl.H ₂ O	11.3, 11.5*	11.4	11.0
KC ₂ H ₃ O ₂	20.2, 20.5*	20.4	19.5
MgCl ₂ .H ₂ O	32.1, 31.9*, 31.7, 32.2*, 32.0	32.0	31.9
Ca(NO ₃) ₂	47.5, 47.4*	47.5	46.8
NaCHO ₂	55.1*, 55.9, 55.6*	55.5	
NH ₄ NO ₃	59.1, 59.3*, 59.2, 59.5*	59.3	59.7
NaCl	74.9, 75.2*, 74.8	75.0	75.1
KCl	83.7, 83.4*, 83.7	83.6	84.5
BaCl ₂ .2H ₂ O	91.2, 91.0*, 91.3	91.2	89.9
KNO ₃	93.0, 93.0*, 93.9, 93.8*, 93.6, 93.4*	93.5	
K ₂ SO ₄	97.9, 97.7*, 97.5, 97.8*, 97.4, 97.5*	97.6	96.8
KClO ₃	98.0, 98.4*, 98.2, 98.0*	98.1	97.7
H ₃ BO ₃	99.2*, 99.0, 98.8*	99.0	

* Values marked with asterisk denote runs where the salt saturator preceded the water.

TABLE III
Coal Samples Tested

<u>Sample No.</u>	<u>Source</u>	<u>Classification</u>
<u>Alberta Coals</u>		
	<u>Area</u>	
2-40	Castor	Subbituminous C
3-40	Castor	Subbituminous C
4-40	Camrose	Subbituminous C
5-40	Camrose	Subbituminous B
6-40	Camrose	Subbituminous B
7-40	Camrose	Subbituminous C
8-40	Tofield	Subbituminous C
9-40	Camrose	Subbituminous C
10-40	Tofield	Subbituminous C
11-40	Tofield	Subbituminous C
12-40	Edmonton	Subbituminous C
13-40	Redcliff	Subbituminous C
14-40	Redcliff	Subbituminous C
16-40	Pakowki	Subbituminous C
17-40	Castor	Subbituminous C
18-40	Camrose	Subbituminous C
19-40	Camrose	Subbituminous C
20-40	Castor	Subbituminous C
21-40	Castor	Subbituminous C
22-40	Sheerness	Subbituminous C
23-40	Sheerness	Subbituminous C
24-40	Sheerness	Subbituminous C
25-40	Castor	Subbituminous C
26-40	Castor	Subbituminous C
27-40	Camrose	Subbituminous C
30-40	Pakowki	Subbituminous C
31-40	Pakowki	Subbituminous C
32-40	Pakowki	Lignite
33-40	Edmonton	Subbituminous B
34-40	Halcourt	Subbituminous A
35-40	Halcourt	Subbituminous A
36-40	Gleichen	Subbituminous A
<u>Saskatchewan Coals</u>		
	<u>Locality</u>	
201-39	Roche Percee	Lignite
202-39	Pinto	Subbituminous C
203-39	Estevan	Lignite
204-39	Taylorlton	Lignite
205-39	Estevan	Lignite
206-39	Estevan	Lignite
207-39	Bienfait	Lignite
208-39	Estevan	Lignite
209-39	Bienfait	Lignite
210-39	Roche Percee	Lignite
211-39	Taylorlton	Lignite

TABLE III - continued

Sample No.	Source	Classification
<u>U.S.A. Coals</u>		
	<u>State</u>	
212-39	Washington	Subbituminous C
213-39	Washington	Lignite
214-39	Washington	Lignite
201-40	Colorado	Subbituminous B
202-40	Colorado	Subbituminous B
203-40	Colorado	Subbituminous B
204-40	Wyoming	Subbituminous A
205-40	Wyoming	Subbituminous B
206-40	Montana	Subbituminous B
207-40	North Dakota	Lignite
208-40	North Dakota	Lignite

"moist basis" and for which a determination of capacity moisture is necessary.

The procedure followed was the same as that used by Stansfield and Gilbert, already described. Vacuum desiccators and controlled humidities were employed, and the determined retained moisture values plotted against the corresponding relative humidities.

In the first 46 determinations the salts used were lithium chloride, potassium acetate, calcium nitrate, ammonium nitrate, sodium chloride, potassium sulphate, and potassium chlorate. In the last 8 determinations, potassium acetate, calcium nitrate, ammonium nitrate, sodium chloride, potassium chloride, barium chloride, potassium nitrate, potassium sulphate and boric acid were used. In plotting the curves, the averaged values given in table II were employed.

The primary object of the work was to check the accuracy of the assumption that the retained moisture over the saturated potassium sulphate solution, for all coals, approximates 98.6% of the capacity moisture found by extrapolation of the moisture-humidity curve. (As previously stated this

value had been challenged by Nicolls, particularly as applied to high moisture coals).

Accordingly the retained moisture over the potassium sulphate solution for each coal was calculated as a percentage of the capacity moisture as determined from the corresponding curve. As a further check, the capacity moisture for each coal was calculated by multiplying the potassium sulphate retained moisture value by $\frac{100}{98.6}$, and the values thus obtained were compared with the corresponding capacity moisture values from the curves.

Sample Determination. The values determined for one of the coals is given as follows:

Coal sample No. 12-40

Source - Edmonton area.

Determined fixed carbon calculated on the dry, mineral-matter-free basis = 58.6%

B.t.u./lb. calculated on the capacity moisture mineral matter-free basis = 9,300

Classification = Subbituminous C.

<u>Saturated salt</u>	<u>Relative Humidity %</u>	<u>Retained Moisture %</u>	<u>Average</u>
Lithium chloride	11.4	(1) 5.5 (2) 5.5	5.5
Potassium acetate	20.4	(1) 7.9 (2) 7.9	7.9
Calcium nitrate	47.5	(1) 17.0 (2) 17.2	17.1
Ammonium nitrate	59.3	(1) 20.4 (2) 20.5	20.5
Sodium chloride	75.0	(1) 23.0 (2) 22.8	22.9
Potassium chloride	83.6	(1) 24.1 (2) 24.1	24.1
Potassium sulphate	97.6	(1) 25.9 (2) 25.9	25.9
Potassium chlorate	98.1	(1) 26.2 (2) 26.2	26.2

Capacity moisture from extrapolated curve (see fig. III) = 26.3%

Capacity moisture, calculated from the potassium sulfate value = $25.9 \times \frac{100}{98.6}$ = 26.3%

Percentage $\frac{\text{Potassium sulphate value}}{\text{Capacity moisture from curve}} = \frac{25.9 \times 100}{26.3} = 98.5\%$

Determined Values. The retained moisture values determined for each sample, together with the capacity moisture values determined by extrapolation, are shown graphically in figures III to IX inclusive. These capacity moisture values are listed in table IV, (column 3), and compared with the values obtained by calculation (column 5). The retained moisture of each coal over the potassium sulphate solution, expressed as a percentage of the corresponding capacity moisture from the curves, is shown in column 4.

The samples in tables and charts are arranged in order of descending capacity moistures for each of the divisions, Alberta coals, Saskatchewan coals and U.S.A. coals.

In order to illustrate the general trend of the moisture curves, discussed later, a composite table was compiled, using the values obtained in the current investigation together with values from previous moisture determinations made by Gilbert. The whole list of values from 119 coals was arranged in order of increasing capacity moistures, and grouped as nearly as possible in representative ranges. The retained moisture values for each coal at stated humidities of 20, 40, 60, 80, 90 and 97 percent, as shown by its curve, were expressed as percentages of the capacity moisture. These percentages for each group were then averaged, and these average values plotted against the corresponding humidities.

The averaged values are given in table V and the curve for each group shown in figure X.

The grouped averaged values for the potassium sulphate factor are also included in this table.

Discussion of Results. An examination of table IV shows the accuracy of the calculation method for determining capacity moistures. In only

FIGURE III

RETAINED MOISTURE - HUMIDITY
CURVES

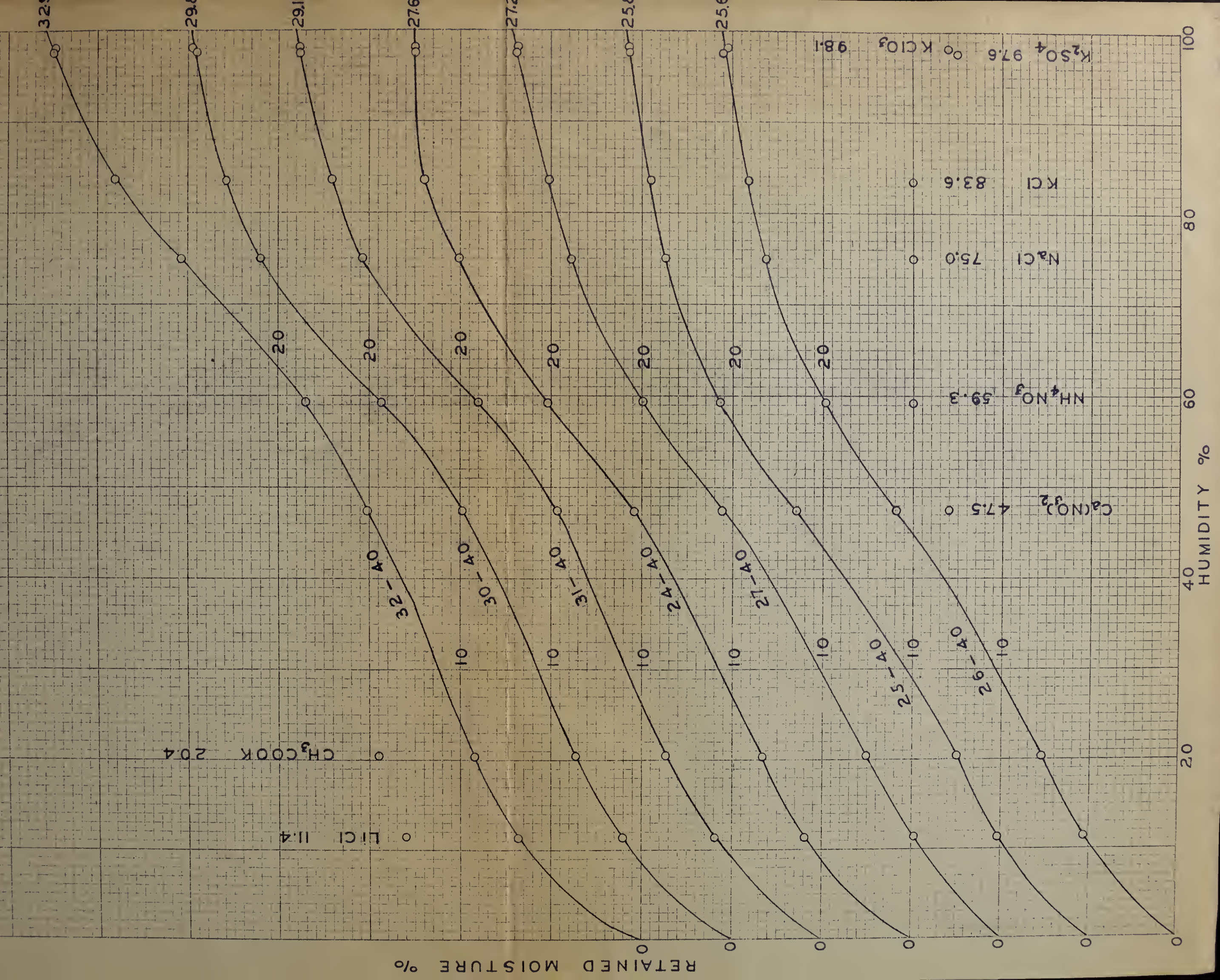


FIGURE IV

FIGURE V.

RETAINED MOISTURE - HUMIDITY
CURVES

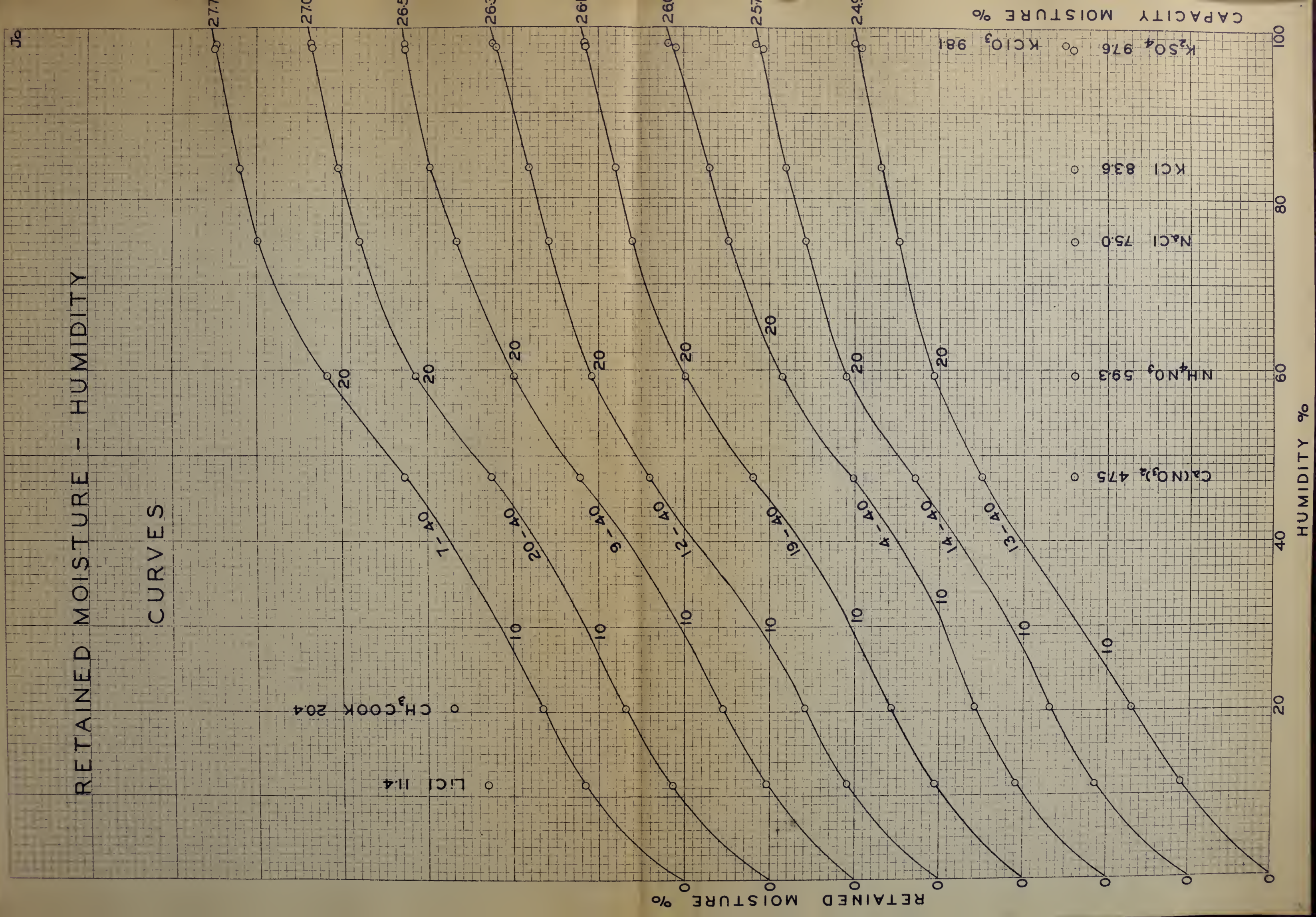


FIGURE VI.

RETAINED MOISTURE - HUMIDITY CURVES

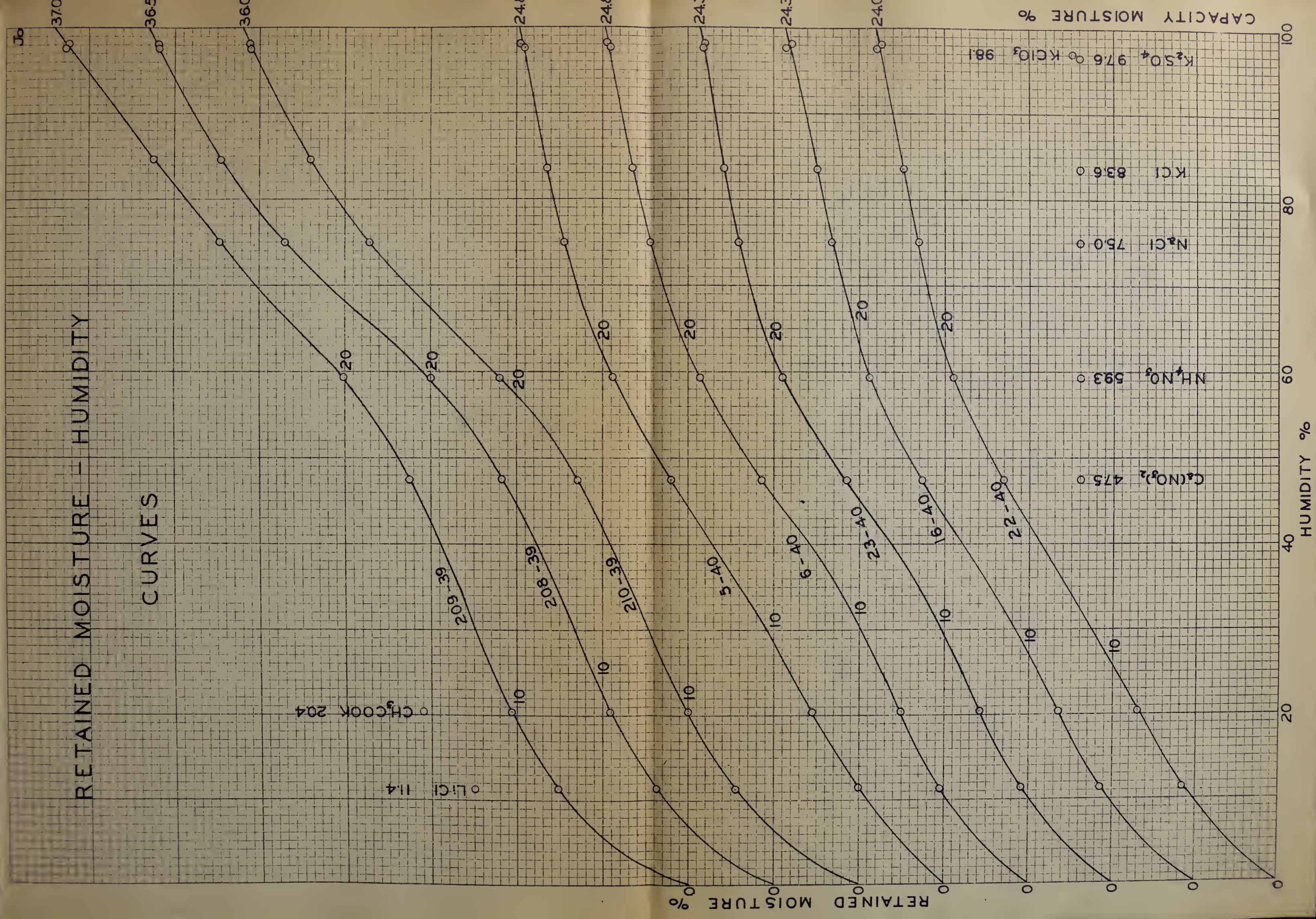


FIGURE VII.

RETAINED MOISTURE - HUMIDITY CURVES

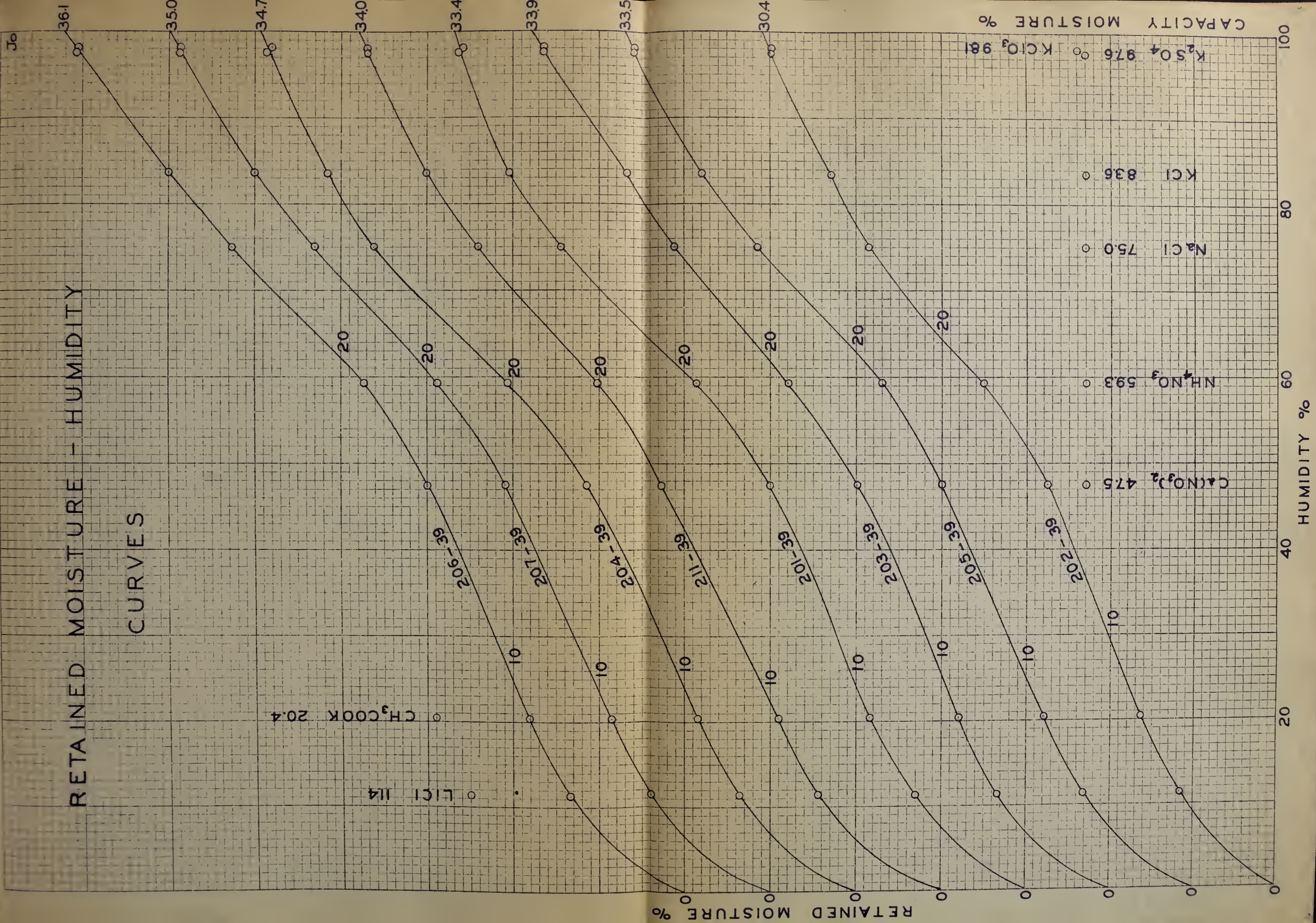


FIGURE VIII.

FIGURE IX

TABLE IV

Capacity Moisture Comparisons

(1)	(2)	(3)	(4)	(5)	(6)	(7)
Sample No.	Retained moisture over K_2SO_4	Capacity moisture from curve	Factor % $(2) \div (3)$	Cap. moist. calculated $(2) \times \frac{100}{98.6}$	Difference $(5) - (3)$	Figure
	%	%	%	%	%	
<u>Alberta coals.</u>						
32-40	32.5	32.8	99.1	33.0	0.2	III
21-40	30.0	30.3	99.0	30.4	0.1	IV
3-40	29.9	30.1	99.4	30.3	0.2	IV
30-40	29.5	29.8	99.0	29.9	0.1	III
2-40	28.9	29.2	99.0	29.3	0.1	IV
31-40	28.8	29.1	99.3	29.2	0.1	III
18-40	28.4	28.7	99.0	28.8	0.1	IV
17-40	28.2	28.4	99.3	28.6	0.2	IV
11-40	27.8	28.1	99.0	28.2	0.1	IV
10-40	27.6	27.8	99.3	28.0	0.2	IV
8-40	27.4	27.7	99.0	27.8	0.1	IV
7-40	27.6	27.7	99.6	28.0	0.3	V
24-40	27.5	27.6	99.6	27.9	0.3	III
20-40	26.8	27.0	99.3	27.2	0.2	V
27-40	26.9	27.2	99.0	27.3	0.1	III
9-40	26.4	26.5	99.6	26.8	0.3	V
12-40	25.9	26.3	98.5	26.3	0.0	V
33-40	25.9	26.2	98.9	26.3	0.1	IX
19-40	25.8	26.1	98.9	26.2	0.1	V
4-40	25.4	26.0	97.7	25.8	-0.2	V
25-40	25.7	25.8	99.6	26.1	0.3	III
14-40	25.3	25.7	98.5	25.7	0.0	V
26-40	25.5	25.6	99.6	25.9	0.3	III
13-40	24.5	24.9	98.4	24.8	-0.1	V
5-40	24.6	24.8	99.2	24.9	0.1	VI
6-40	24.6	24.8	99.2	24.9	0.1	VI
23-40	24.2	24.3	99.6	24.5	0.2	VI
16-40	24.2	24.3	100.0	24.6	0.3	VI
22-40	23.9	24.0	99.6	24.2	0.2	VI
36-40	17.5	17.8	98.3	17.8	0.0	IX
35-40	16.2	16.3	99.4	16.5	0.2	IX
34-40	15.5	15.8	98.1	15.7	-0.1	IX

Table IV - continued.

(1)	(2)	(3)	(4)	(5)	(6)	(7)
<u>Saskatchewan coals.</u>						
209-39	36.4	37.0	98.4	36.9	-0.1	VI
208-39	35.9	36.5	98.4	36.4	-0.1	VI
206-39	35.3	36.1	97.8	35.8	-0.3	VII
210-39	35.6	36.0	98.9	36.1	0.1	VI
207-39	34.4	35.0	98.3	34.9	-0.1	VII
204-39	34.3	34.7	98.8	34.8	0.1	VII
211-39	33.6	34.0	98.8	34.1	0.1	VII
203-39	33.2	33.9	98.0	33.7	-0.2	VII
205-39	33.0	33.5	98.5	33.5	0.0	VII
201-39	33.2	33.4	99.4	33.7	0.3	VII
202-39	30.0	30.4	98.7	30.4	0.0	VII
<u>U.S.A. coals</u>						
207-40	39.5	41.0	96.3	40.0	-1.0	IX
208-40	33.3	34.3	96.8	33.8	-0.6	IX
213-39	32.8	33.5	97.9	33.3	-0.2	VIII
214-39	31.5	31.9	98.8	32.0	0.1	VIII
212-39	28.8	29.0	99.3	29.2	0.2	VIII
206-40	25.4	25.9	98.1	25.7	-0.2	IX
205-40	24.4	24.7	98.8	24.7	0.0	IX
201-40	23.7	24.0	98.8	24.0	0.0	VIII
203-40	23.2	23.5	98.7	23.5	0.0	VIII
202-40	18.2	18.4	98.9	18.5	0.1	VIII
204-40	11.9	12.1	98.4	12.1	0.0	VIII

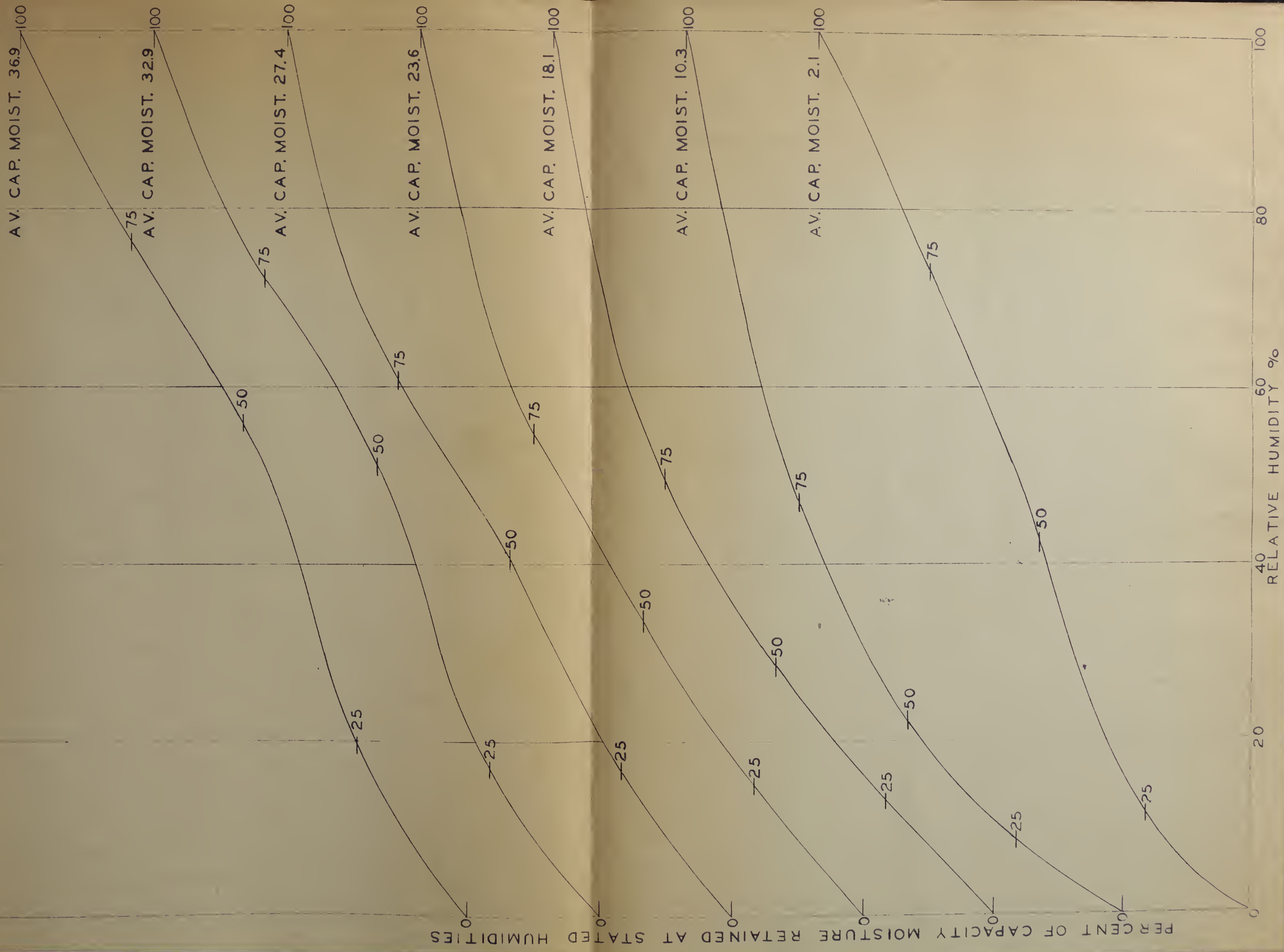
two instances is the difference between the calculated and extrapolated values greater than 0.3%, and these are of exceptionally high moisture coals.

A study of table V shows slight confirmation of Nicolls' claim that the potassium sulphate factor is lower for high moisture coals, as in this table the value for coals from 30-35% moisture averages 98.2 and for coals from 35-41% moisture averages 98.0. However, the average value of 98.8 for the 54 high moisture coals tested in the current investigation, shown

FIGURE X

Composite curves of the grouped values of retained moistures expressed as percentages of the capacity moisture at stated humidities.





in column 4 table IV, lends no support to Nicolls suggestion that for general use the value 98.6 previously used be reduced to $97.1 \frac{(100)}{1.03}$.

Table VI gives a comparison of moisture values determined in Edmonton, Ottawa and Pittsburgh. It will be noted that the Edmonton values tend to be a little higher than the others, but in general they agree reasonably closely, particularly in consideration of the high moisture content of most of the samples.

It might be noted that there is no officially adopted standard for the determining of retained moisture in coarsely ground coals. In the Pittsburgh laboratories, moisture determinations are known to be made in an oxidizing atmosphere, while in Edmonton a neutral atmosphere is used. The latter gives higher moisture values.

Some interesting features may be seen in the curves. The lower moisture coals have simple curves, while the high moisture coals have distinctly double curves, with a gradual change from the former to the latter as the moisture content of the coals increases. This is more clearly shown by the composite curves of figure X.

In the lowest composite curve the capacity moistures are all below 4%. The retained moistures, expressed as percentages of these low capacity moistures, are therefore liable to large numerical errors, and no great significance can be attached to this curve. It might also be noted that all the coals in this group are bituminous coals from the Kootenay geological horizon, and that no similar coals are included in the other groups.

Several explanations of the double curvature of the high moisture coals might be submitted. One suggestion is that it is caused by the heterogeneous character of the coal, but this is improbable. Another suggestion is that the curve is characteristic of the manner in which moisture is held

TABLE V

Grouped Values of Retained Moistures as % of Capacity
Moisture at Stated Humidities

No. of samples	Range of capacity moisture %	Average capacity moisture %	Percent of capacity moisture retained at stated humidity						Grouped values of K ₂ SO ₄ factor
			20%	40%	60%	80%	90%	97%	
8	1 - 4	2.1	34.2	48.6	63.4	81.0	90.0	97.0	96.0
17	4 - 13	10.3	47.3	68.2	83.0	92.3	96.1	98.6	98.5
21	15 - 20	18.1	36.7	65.7	83.7	93.1	96.5	98.9	98.8
20	20 - 25	23.6	32.4	57.4	79.2	91.7	95.8	98.7	98.8
32	25 - 30	27.4	29.4	49.2	74.0	91.0	95.7	98.8	99.0
15	30 - 35	32.9	28.1	40.9	59.0	84.0	92.5	97.7	98.2
6	35 - 45	36.9	25.8	37.7	54.2	79.5	90.2	97.0	98.0

in the coal. Thus Porter and Ralston (5) suggest that moisture may be held in three ways: adsorbed on the surface, held in capillaries, and closely combined as a gel. This may give an explanation of the double curvature with the high moisture coals, as a double curvature may be noted in the vapor pressure - water content curve of a silica gel. Figure XI is ~~modification of~~ a silica gel curve developed by van Bemmelen ^{modified and} explained by Zsigmondy (9).

A silica gel with high moisture content, if slowly dried, continues to have practically the same vapor pressure as pure water until the moisture content falls to about 6 mols. of water to one mol of silica (65% moisture). This is the point A in the curve. At this point the capillaries and cells are full of water. With a continued drying, from A to B in the curve, the capillaries remain full of water but shrink in size, and the vapor pressure falls. At B the volume change stops, and with further drying

TABLE VI

Comparison of Moisture Values Determined in Edmonton, Ottawa and Pittsburgh. (Deviations from Edmonton Values)

	Moisture as rec.	60% Humidity	75% Humidity	90% Humidity	97% Humidity	100% Extra- polated	100% calculated
<u>Ottawa</u>							
No. of values compared	51	19	41	41	49	47	49
Algebraic average of deviations	-0.4	-0.5	0.0	-1.0	-0.7	-0.5	-0.3
Arithmetic average of deviations	0.6	0.6	1.1	1.0	0.8	0.7	0.6
<u>Pittsburgh</u>							
No. of values compares	18	-	18	18	18	18	18
Algebraic average of deviations	-0.9	-	-0.8	-1.0	-0.8	-0.7	-0.5
Arithmetic average of deviations	1.1	-	0.9	1.0	0.9	0.8	0.6

from B to C in the curve, the moisture in the capillaries is replaced by air, with little change in the vapor pressure. At C the capillaries only contain the strongly held adsorbed water. With further drying this also is removed, as seen from C to D in the curve, with a rapid fall of vapor pressure.

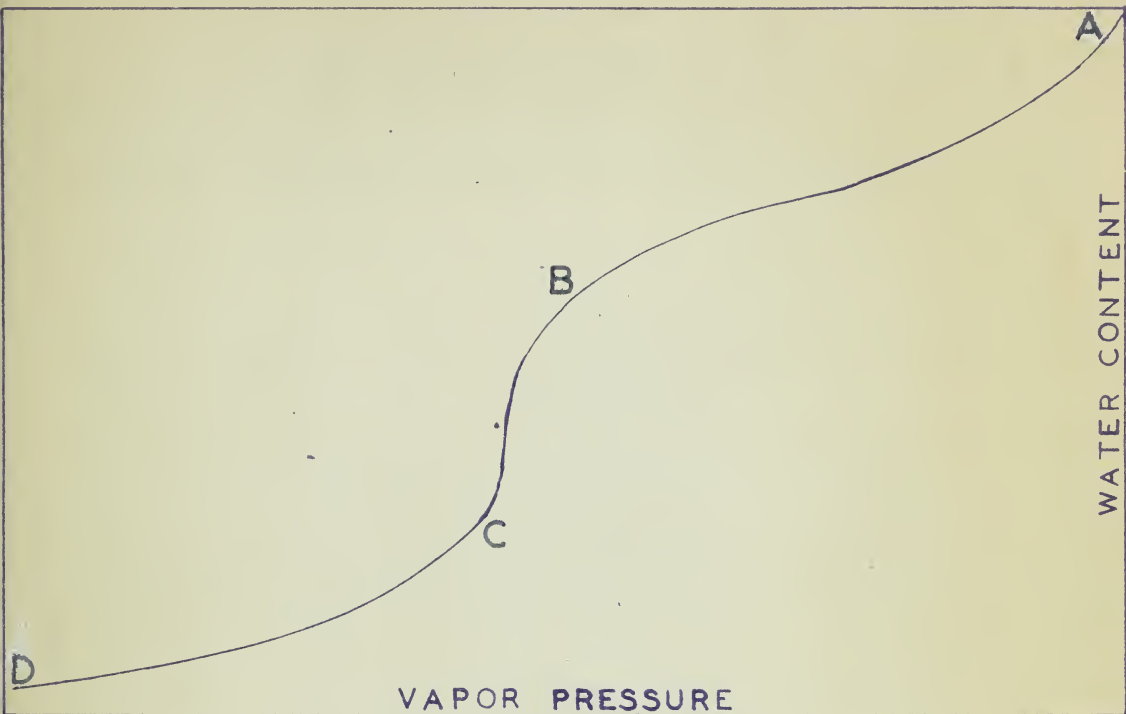
The above explanation is in accord with the changes in transparency in the gel at points B and C.

It is suggested, by analogy, that high moisture coals contain notable amounts of gel, while low moisture coals do not. It might be noted that the point A in the above curve corresponds to the capacity moisture point for coals.

Time did not permit further study of this matter.

FIGURE XI

The vapor pressure - water content curve of a
silica gel.



The investigation of the various salt solutions and of their use in the determination of capacity moisture, showed that potassium sulphate can be satisfactorily employed for a high humidity.

Boric acid has proved quite satisfactory in the tests made, and has the further advantage that its higher humidity (99%) would reduce the errors in computing capacity moisture. Until further tests have been made with boric acid, however, the potassium sulphate values will be used as before.

C H A P T E R I I I
W E T T I N G A G E N T S F O R C O A L D U S T

1. Introduction.

One of the serious problems in coal mining is the prevention of dust explosions. Even with modern precautionary measures the danger of explosions must still be recognized (10) and various means have been adopted to prevent them. Rock dusting has long been recognized as an efficient preventative measure (11) but this process tends to aggravate the general dust condition. Spraying with water is a simple means of decreasing the dust hazard, and is advocated by Harrington and Forbes (12) as a measure for "forwarding both health and safety". Hartmann and Greenwald (13) in later investigations, suggest the use of both rock dusting and spraying, in that one supplements the other. However, water dries out quickly, and its protection is lost. Also, bituminous coal dust, which is commonly explosive, is notably difficult to wet.

The above suggests the importance to the coal mining industry of methods for wetting coal dust. Tideswell and Wheeler (14) investigated certain wetting agents for use in spraying in mines, but the materials with which they worked were not available in Canada and their investigation left several questions unsettled.

An investigation of wetting agents was thought advisable for the added reasons that it might assist in the laboratory problem of determining the specific gravity of coal, and also might possibly be used in methods for evaluating the fineness of grinding.

2. Materials.

Canadian Chemical Industries were approached and submitted for consideration the following six samples of commercial wetting agents, together with descriptive literature.

Alkanol B. A light yellow powder, readily soluble, described as the sodium salt of a naphthalene sulphonic acid derivative.

Alkanol S.A. Described as a less purified form of Alkanol B.

Dupanol W.A. White flakes. Probably a sulphated higher fatty alcohol.

Perminol W. A brown powder. No description was provided.

Lissapol L.S.Paste. A grayish white paste. Stated in the literature as not being a sulphated fatty alcohol.

Neopen S.S. A brown powder. Described as the sodium salt of abietene sulphonic acid.

3. Experimental Tests.

Surface Tension. A quantitative determination of the surface tension of various concentrations of solutions of the agents was carried out. A Traube stalagmometer was used and the measurements made by the comparative drop method with water as the standard. Temperature was controlled at 20-25°C with the standard surface tension of water taken as 72.5 dynes per cm.

It might be assumed that the greater the reduction in surface tension, from that of pure water, the better the wetting power. Hence the salts are listed in table VII in order of descending reduction (at 1% concentration). ^{They are} ~~shown~~ shown graphically in figure XII.

From an examination of figure XII it is seen that Dupanol W.A. lowers the surface tension to the greatest extent, and with the greatest effect at low concentrations. The curves show that concentrations greater than 0.5% have little added effect on the surface tension. The graph also

TABLE VII

Surface Tension of Wetting Agents - Dynes per cm.

Materials	Dupanol WA	Alkanol B	Alkanol SA	Lissapol Paste	PerminolW	Neopen SS
Concentration %						
1	30.5	32.6	33.2	34.6	35.4	38.7
0.75	31.8	33.1	33.7	37.0	37.4	39.0
0.5	32.6	35.3	34.8	38.4	40.1	41.0
0.4	32.4	37.6	36.7		41.5	
0.3	33.1	40.9	40.5	39.9	44.6	44.5
0.2	36.0	46.0	45.0		51.3	
0.1	40.5	54.5	52.7	47.5	60.7	53.8
0.075	46.3		57.5		63.2	
0.05	49.2		62.4		67.8	
0.02	61.2		71.2		72.5	

indicates the similarity of Alkanol B and Alkanol S.A.

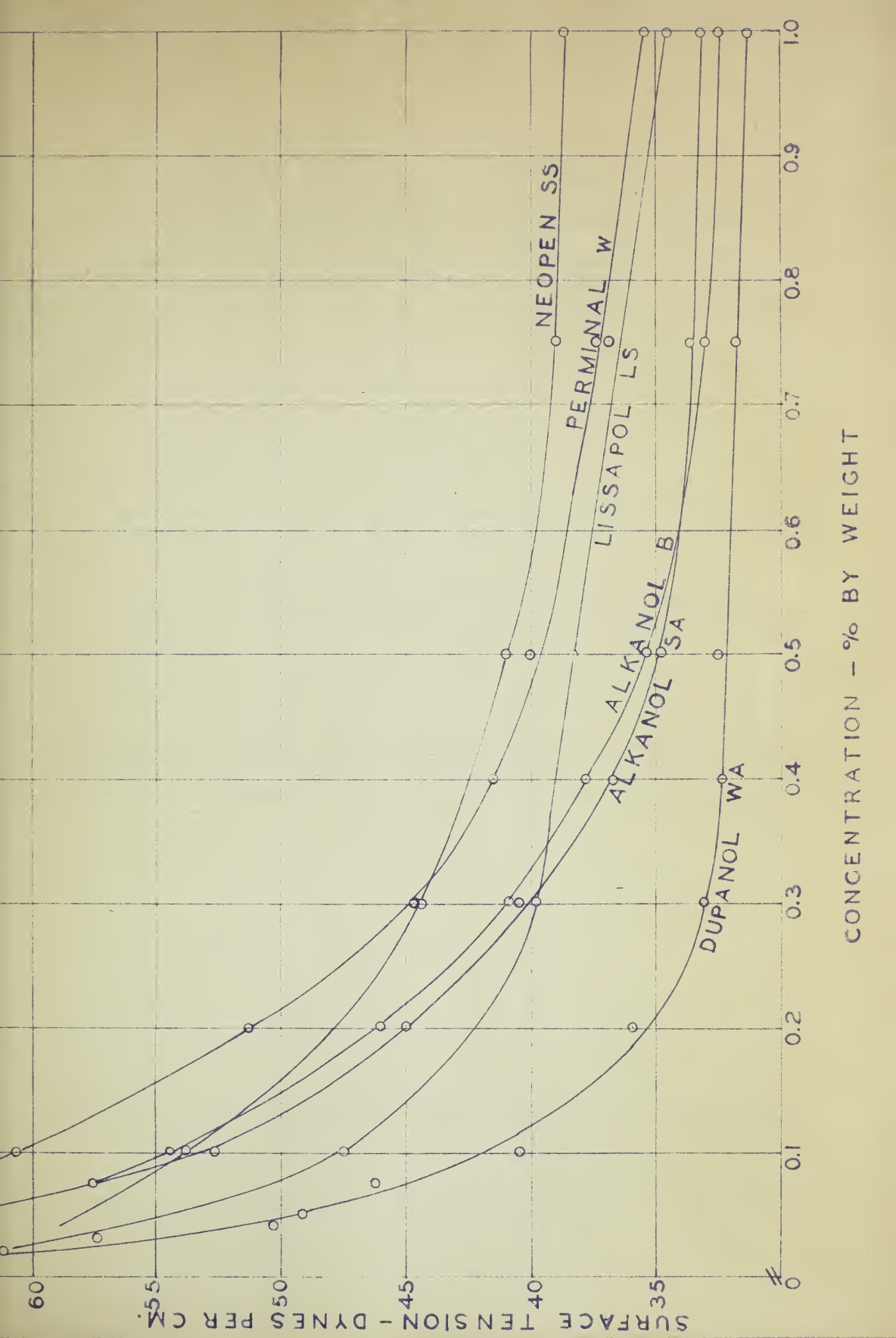
Rate of Wetting. A determination was made of the comparative rates of wetting of the six materials. Throughout the tests one sample of coal dust was used. This was prepared from a coking bituminous coal from the Hill-crest collieries, very difficult to wet, having a low moisture content (of approximately 1.5%) and a high volatile matter content (of approximately 28%.)

The sample was ground in a ball mill for two hours, and gave a screen analysis of 98% through 150 mesh, and 94.5% through 200 mesh.

In a later test a sample of coal dust from Brazeau was used for comparison of coals. This had a moisture content of approximately 1.5%, a volatile matter content of 16% and a screen analysis of 97% through 150 mesh and 90% through 200 mesh.

FIGURE XII

Surface Tension - Concentration curves of
wetting agents.



The test used was simple. The solution to be tested was placed in a one inch diameter test tube and a small scoopful of dust (approx. 0.055 g.) was dusted through a screen onto the surface. The time taken for the dust to be wetted and sink was measured with a stop watch.

Ten or twelve determinations were made on each concentration of each wetting agent. The averaged results are shown in table VIII with the salts listed in the same order as in table VII.

A blank determination was made, using distilled water, with no wetting observed during a period of 4 days.

An example of a set of determinations showing the agreement of check results, is given as follows:

Solution of Dupanol W.A. Concentration = 0.75% by weight
 Time of wetting in seconds: 29,38,35,40,32,39,39,36,32,36,35,34.
 Average = 35 seconds

TABLE VIII

Rate of Wetting of Coal Dust

Wetting Agent	Dupanol W.A.	Alkanol B	Alkanol S.A.	Lissapol Paste	Perminol W.	Neopen S.S.
Concentration %				Time in Seconds		
3				390	20	900
2					30	1800
1	25	60	30	3600+	155	3600
0.75	35	85	50		375	
0.50	85	390	215		1800	
0.30	170					

From the data of table VIII the materials were arranged in order of rate of wetting as follows: 1-Dupanol W.A., 2,-Alkanol S.A., 3-Alkanol B, 4-Perminol W, 5-Neopen S.S., 6-Lissapol L.S.Paste.

This order, except for Lissapol L.S. Paste, is in agreement with the surface tension measurements as shown in figure XII.

Quantitative Tests of Wetting. Preliminary tests, made by pipetting 1 ml. of a given solution into a test tube containing 5 grams of coal dust, and determining the quantity of dust wetted after 24 hours, proved unsatisfactory. However, the results of the tests gave a number of helpful indications. It appeared, first, that the wetting material was adsorbed ~~and~~ by the coal, and thus removed from the solution, as the top layers of the coal dust would be readily wet while the solution failed to penetrate to the lower layers; second, that the wetting materials which were more readily adsorbed did less actual wetting in the time allowed for the test than those less readily adsorbed; third, that calculated on a basis of grams of coal wetted per gram of material used, the solutions of lower concentration gave the higher results. This suggests that an unnecessarily high concentration of adsorbed substance on the surface of the top layers of the dust results from the use of the more concentrated solutions.

Bearing in mind the above indications, a satisfactory test was developed and employed as follows.

A spraying chamber, 25 inches by 10 inches and 10 inches deep was constructed with a removable cover and a glass observation window. The spray nozzle was inserted at one end, with the jet 4-5 inches from the end and 0.5 inches from the top. A layer of coal dust, ^(about 1g) about 1 mm. in thickness was sprinkled onto a petri dish 7 cm. in diameter, and the dish weighed with its cover. The dish without the cover, but with a guard, was then placed on a stand $2\frac{1}{2}$ inches above the floor of the chamber. An inch or so of water on the floor maintained 100% humidity in the chamber.

The solution was sprayed horizontally into the chamber and settled

onto the dust in the petri dish until small droplets covered the surface quite thickly (0.3-0.5 g.). A guard cover protected the walls of the dish from being wet, and only the dust received the spray. The dish was then removed, covered, weighed to measure the quantity of deposited spray, and placed in another humidity chamber containing a reservoir of water. This in turn was placed in a constant temperature chamber at 30°C. The last two precautions were taken so that neither evaporation of the spray nor condensation of dew would spoil the experiment.

After standing 24 hours, the contents of the dishes were washed into beakers. The unwetted coal floated on the surface and was skimmed off. The wetted dust was collected by filtering through a weighed gooch crucible with an asbestos pad. The crucible was then dried in vacuo at 100°C to constant weight and the dry weight of the wetted dust thus determined.

A blank determination was made with no spraying to determine how much dust was wet by water during the operations of washing out the petri dishes and the skinning off of the unwetted dust. A correction was then calculated for each determination on the basis of the grams of unwetted dust handled. The results were computed to a basis of grams of dust wetted per gram of wetting agent, thus reducing the results for all concentrations to a comparable basis.

The above spraying method aimed at securing a maximum surface exposure to avoid the errors of adsorption mentioned in the preliminary tests.

A series of determinations was run in triplicate, using all the wetting agents at various concentrations, with the averaged results shown in table IX.

The same coal dust was used for this series of tests as was used in the "rate of wetting" tests. As a comparison of coals, the sample of Brazeau coal dust previously mentioned was tested, with the results in-

cluded in table IX.

An example of a determination is given as follows:

Wetting agent - Alkanol B.

Concentration 1%

	1	2	3	3 ÷ 2
Wt. of spray grams	Wt. of wetting agent (dry) grams	Wt. of dust wetted grams	Grams of dust wet per gram of material	
(1)	0.494	0.00494	1.363	276
(2)	0.355	0.00355	1.012	285
(3)	0.560	0.00560	1.476	264
		Average		275

TABLE IX

Quantities of Coal Dust Wetted

Concentration Wetting agent	1%	0.5%	0.3%
	(Grams of coal wetted per gram of material)		
<u>Hillerest coal dust.</u>			
Alkanol B	300	295	
Neopen S.S.	250	270	
Dupanol W.A.	240	265	245
Alkanol S.A.	230	225	275
Perminol W.	135	130	
Lissapol L.S.Paste	90	85	
<u>Brazeau coal dust.</u>			
Dupanol W.A.	275	300	
Alkanol S.A.	260	275	

and above

Table IX_A shows, first, that reasonably consistent results can be secured by this test; second, that within the limits tested, concentration has little effect on the quantity of dust wetted for a given quantity of wetting agent; third, that Perminol W and Lissapol L.S. Paste are distinctly inferior as wetting agents for coal dust, while Alkanol B appears

to be the best.

A comparison of the results with the Hillcrest and Brazeau coal dusts indicates that similar results are obtained with quite different coals, and that the wetting agents act equally well on coals of high and of low volatile matter content.

Adsorption Tests. In order to demonstrate the adsorption of the wetting materials by the coal from the solution, the following determinations were made.

(A) A measured quantity of 10 ml. of 1% Dupanol solution was placed in a beaker on a rotating table, and successive 1 gram portions of dust added. The time taken for each additional portion to be wetted was measured by a stop watch. The tests were made in duplicate.

(B) A series of beakers, each containing 10 ml. of 1% Dupanol solution, was set up and coal dust was added to each, one gram to the first, two grams to the second, and so on. These were placed on the rotating table and when wetting was complete, each solution was filtered and a surface tension determination made on the filtrate.

It was found that the filtering of the solutions allowed a notable adsorption of wetting agent by the filterpad, giving noticeably higher surface tension measurements than shown by 1% Dupanol in table VII.

In (A) the first gram added gave erratic results, possibly due to uneven stirring.

The results are given in table X and shown graphically in figure XIII.

A definite similarity is seen in the two curves of figure XIII, indicating that the increase in wetting time was due to the increase in surface tension. This presumably resulted from a decrease in the concentration of the solution due to the adsorption of the wetting agent by the coal dust.

TABLE X

Surface Tension Related to Time of Wetting.

Grams of dust wetted	A. Wetting time per gram added (minutes)	B. Surface Tension Dynes per'cm.
1	-	35.5
2	1.1	35.5
3	1.1	35.5
4	1.1	35.6
5	1.1	35.4
6	1.0	35.5
7	1.5	35.6
8	2.0	35.8
9	2.2	36.3
10	4.1	37.1
11	5.9	39.2

Rewetting. A further evidence of the adsorption of the wetting agents by the coal dust was given by rewetting tests. Portions of the Hillcrest coal dust that had previously been wetted with 1% solutions of Dupanol W.A., Alkanol S.A. and Perminol W, were dried, exposed in the laboratory for a week and then tested for "rate of wetting" as previously described, but with distilled water in the test tubes. The time of wetting was as follows: Dupanol W.A. 25 seconds, Alkanol S.A. 50 seconds, Perminol W. 60 seconds. These results when compared with those of table VIII show that dust, previously wetted with agents and dried, is rewetted at the same rate by pure water as ^{is} untreated dust by 1% solutions.

This was in accordance with the findings of Tideswell and Wheeler (14).

Corrosion. In view of the fact that use of such agents in mine sprinklers might result in corrosion of rails, tools, etc., the corrosive action of 3% solutions of each of the wetting agents on iron was observed. After a month's contact the corrosion shown was negligible.

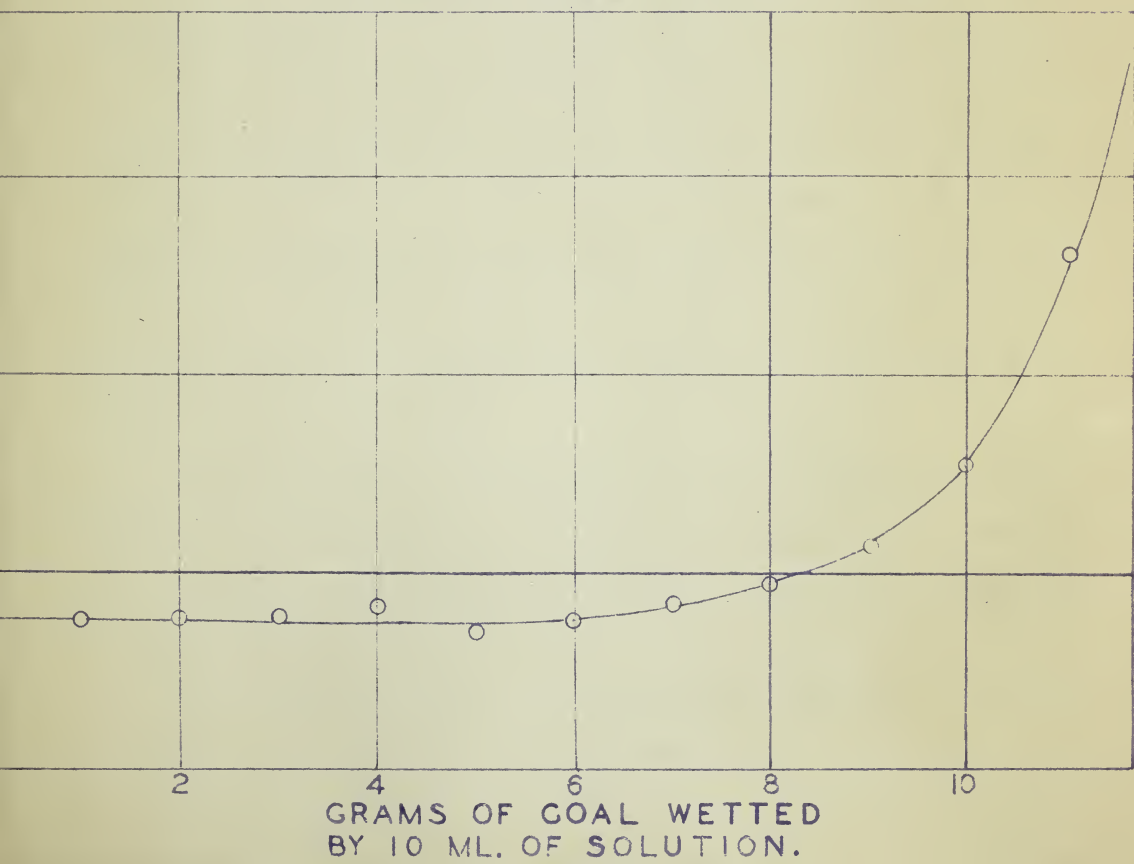
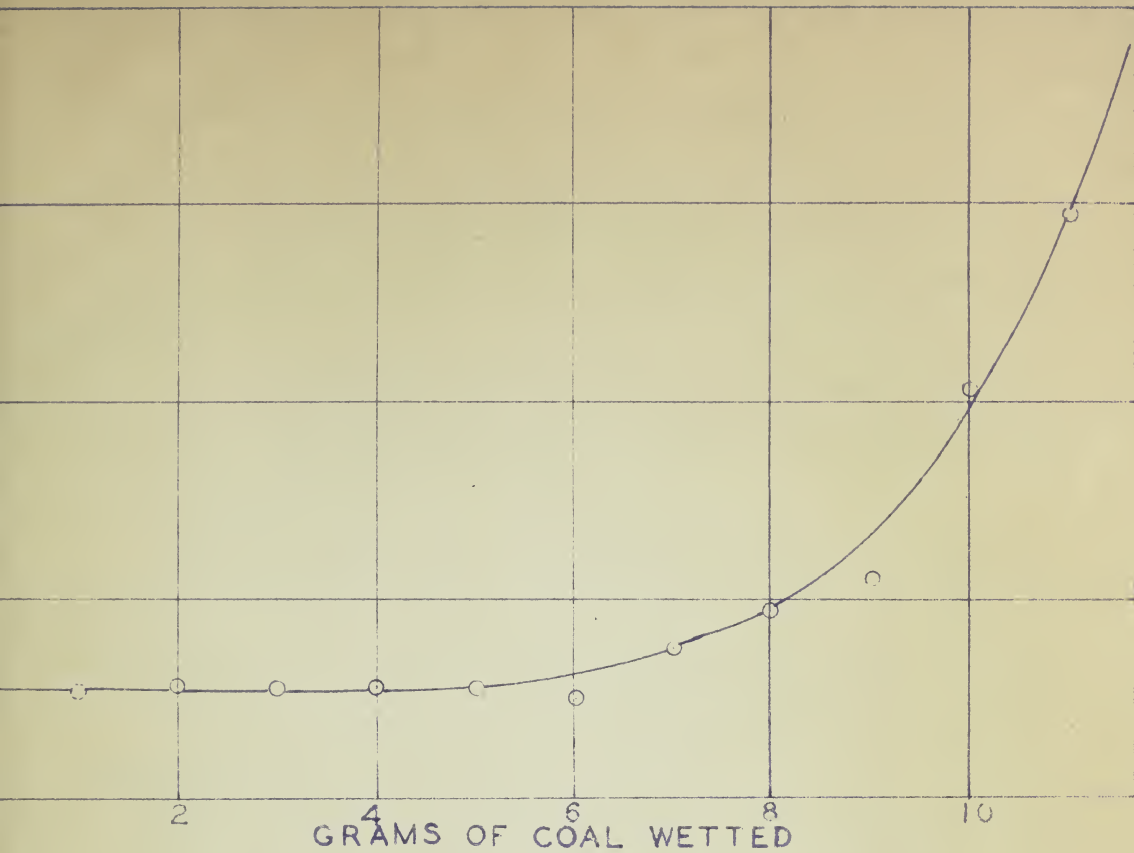
Discussion of Results.

The tests indicate that of the six substances tested, four are suit-

FIGURE XIII

Relation of Wetting Time and Surface Tension Change.

(10 ml. of 1% Dupanol, and Hillcrest coal dust).



able for coal wetting; namely, Alkanol B, Neopen S.S., Alkanol S.A., and Dupanol W.A. For rapid and immediate wetting Dupanol W.A. is the best, but it is most readily adsorbed from solution and therefore will not wet as much dust as some of the other agents which are adsorbed less readily.

Table IX indicates that for the coal dusts used and for the concentrations of wetting agents tested, the amount of wetting was directly proportional to the amount of wetting agent.

It might be noted that of the numerous commercial wetting agents now available (15), the six here tested were the ones suggested by Canadian Chemical Industries as being probably best suited for wetting coal dust.

The following suggestions are made for the use of these wetting agents in mines.

First, a solution of 0.5%, or even less, would seem to be a suitable concentration.

Second, after a primary thorough spraying had been carried out in the mine, subsequent sprayings might be done with more dilute solutions. This seems preferable to subsequent spraying with water alone, since later deposits of unwetted dust must normally be dealt with. Thus the wetting material might be adsorbed from the dilute solution by the fresh dust, but the water which was left would readily rewet the previously wetted dust below the fresh surface layer.

Third, for practical application in a mine, the concentration of solution used, and the frequency and quantity of spraying, will depend on the conditions as found in the mine, such as: - fineness of dust, presence of dirt, and wettability of the coal.

It is interesting to note that Wheeler (14) suggested the use of a 3% solution of the materials which he tested.

In regard to frequency of spraying, Wheeler's practical tests indicated that for haulage roads one spraying with the solution was adequate for as long as a year, with water sprays at intervals as necessary.

If the mine conditions are such that there is a continual deposit of fresh coal dust, it is suggested that more frequent sprayings with a dilute solution would be better than Wheeler's infrequent spraying with a comparatively strong solution.

Hartmann and Greenwald (13) conducted an investigation of the use of wetting agents in coal mines, but their work was not published until after the work in this laboratory had been completed. They suggest that the use of wetting agents alone cannot effectively prevent mine explosions, but that their use greatly prolongs the effective life of a given application of rock dust. They also suggest the working face of the mine as the most important point for spray application. They used 1% solutions for their laboratory tests, but suggested no particular strength of solution for mine use.

Considering the experimental results from a commercial point of view a number of points might be noted. At a quoted price of 50 cents per pound, any of the 4 suitable wetting agents would completely wet a ton of minus 200 mesh coal dust at a cost of approximately \$4.00. This would be even less for ordinary mine conditions where the dust is not so fine.

Expressing it in another way, the cost of 100 gallons of an 0.5% solution for spraying would be \$2.50.

It might be noted that at the time this work was carried out in this laboratory, the method for determining the specific surface of dusts, later described, had not been developed. It is suggested that the correlation between the amount of wetting agents adsorbed, and the specific surface of the dust wetted would be of sufficient interest to warrant determination.

The above quotation of 50 cents per pound was dated May, 1938 (f.o.b. Montreal) for Dupanol W. A. A recent quotation dated February 1941, gives a price of 20 cents per pound (f.o.b. Montreal) for the same material.

C H A P T E R I V

μ COAL - ITS PREPARATION AND MEASUREMENT.

Introduction.

" μ coal" is the term applied by Franz Fischer to coal so finely powdered that each of the particles has a diameter of 10 microns (10 μ) or less. It matters little whether " μ coal" is a suitable name or not, but a consideration of very finely ground coal is of evident importance. The decrease of particle size and the attendant increase of surface area when coal is ground, greatly affects many coal problems. Determinations of oxidation, carbonization, inflammability, solubility and so on, are all affected by the particle size of the coal under consideration, and as stated by Rosin (16) "the influence of particle size in fuel technology is so extensive that an exhaustive report would be impossible".

Franz Fischer and others (17) introduced the term " μ coal" in a publication dealing with their work on extraction of coal with organic solvents. They claimed that in ordinary ball mill grinding a lower limit is reached, finer than which the coal cannot be reduced, but with vacuum grinding in a properly constructed ball mill a much finer product can be obtained. This product is their " μ coal".

An investigation was undertaken in the laboratories of the Alberta Research Council to study fine grinding of coal, in the light of the above claims.

As suggested in the general introduction, the work of this inves-

tigation fell into three main divisions, which, though intimately connected, are described in separate sections:

- Section A. The determination of the specific gravity of coal.
- Section B. The evaluation of fineness of grinding.
- Section C. The fine grinding of coal.

S E C T I O N A

THE DETERMINATION OF THE SPECIFIC GRAVITY OF COAL

Introduction.

A determination of the specific gravity of coal is required for practical commercial reasons as well as for laboratory tests. In order to measure the potential output of a coal mine, the mine operator must know the specific gravity of the coal. The specific gravity must also be known for coal washing operations.

For such purposes the "apparent" specific gravity of the coal is determined. This is regarded as the specific gravity of the coal in lumps as it occurs in the seam, and is found by weighing lumps of coal in air and in water. This is of course only an approximate measure of the specific gravity of the coal in the seam, because of the difficulty in securing truly representative lumps of coal.

The determination of the real specific gravity of the coal substance is far more difficult because of the gas present in the cells and cavities of the coal. Various means of dealing with this problem have been tried, with doubtful success. Drakeley and Jones (18) saturated lumps of coal by boiling them in water to remove the gases, but this method obviously is unsatisfactory (19).

Many determinations of both apparent and real specific gravities have been made in the laboratories of the Alberta Research Council, prior to the current investigation. Correlations of the moisture and ash content

to the apparent specific gravity (20) of a large number of coals have been made, but the determinations of the real specific gravity have been somewhat in doubt.

The procedure used was as follows. The coal was ground very finely to liberate most of the gas and the specific gravity found by displacement of water in a pycnometer bottle. The air was removed by boiling, evacuation, or centrifuging. The best results were obtained by pumping all the gas possible out of the coal before the water was admitted, but even so, the method was not satisfactory. Furthermore many coals are difficult to wet by water. Organic liquids were tried in place of water, but with actual or suspected solvent action and change of the coal substance. Kerosene was the best of those tried.

A method has now been developed in these laboratories, using the wetting agents described in Chapter III.

Procedure.

The coal to be tested is finely ground in a ball mill. A solution of a suitable concentration of wetting agent in water is prepared, and its specific gravity determined. Centrifuge tubes of approximately 50 ml. capacity, containing about 25 ml. of the solution, are weighed, about 3 to 4 grams of the finely ground coal added, and the tubes reweighed. The coal is readily wet and sinks to the bottom. The tubes are almost filled with the solution, and are then centrifuged for 10 minutes at approximately 2000 r.p.m. (diameter of swing = 20 inches). This clears ^{the coal from} the solution in the neck of the tube, ~~of coal~~, and tends to force the solution into any of the capillaries of the coal not already filled. After centrifuging the tubes are filled to the mark of known volume and weighed.

The weight of the solution displaced is calculated from the specific

gravity of the solution, the volume of the tube, and the weight of the coal, and from this the specific gravity of the coal is calculated.

For most of the coals tested a concentration of 0.3% solution of Dupanol was found suitable, but for some of the coals more difficult to wet, a 0.5% solution was necessary.

Sample Determination. Lethbridge coal. Solution 0.3% Dupanol (Sp. Gr. 1.001).

	(1) Weight of Coal <u>(grams)</u>	(2) Weight of Solution <u>(grams)</u>	Sp. Gr. of Coal <u>(1) + (2) x 1.001</u>
i	3.669	2.567	1.431
ii	3.138	2.217	1.417
iii	3.277	2.295	1.429

Discussion.

The specific gravity determinations carried out at this time were for use in the evaluation of fineness of grinding, and only three figure accuracy was required. For more accurate determinations a tube with a narrow neck would be required, so that the volume could be more accurately measured.

A number of interesting features were noted in the determinations. When a sample of coarse coal (14-20 mesh) was used, a swelling effect was noted after centrifuging. That is, if the tubes were filled to the mark and left standing for a few hours, the liquid rose above the mark, but on recentrifuging the liquid receded to the mark. This phenomenon was not noted with the finely ground coal. This suggests that the coarse coal still contains considerable gas in the pores and capillaries, which is compressed by centrifuging, and slowly expands on standing.

It was also noted that there was a slight increase in the specific gravity determined on a given sample of coal, with continued grinding. An

ash analysis of portions of the sample taken at intervals of grinding showed that this increase in specific gravity was not due to iron from the balls and mill, as the percentage of ash remained constant. This also would indicate that the finer grinding released more of the gas.

The method was found to be quick and simple, and with some modifications might be suitable for a standard determination of the real specific gravity of coal.

S E C T I O N B

EVALUATION OF FINENESS OF GRINDING

Introduction

As suggested above, the fineness of grinding has a great effect upon various processes and reactions with coal. Therefore the results of experiments with coal must be correlated to an actual measure of fineness. This is especially true with very finely ground or powdered coal (21). Actually it is the surface exposed that is important, but the determination of particle size and surface area may be dealt with as one problem.

Various general methods for determining particle size are here described, with a discussion of their suitability for use with coal.

Screening. This is the universal method for sizing materials (22), but is limited to available sizes of screen openings, and the finest screen ordinarily used is the 200 mesh, with an opening of 0.074 mm. (Tyler Standard). This is not fine enough for experimental work with coal, as the surface area of the sub-screen sizes may far outweigh that of the screened sizes.

Screen analyses of the larger sizes of a ground material may be extrapolated by means of equations to give the size of the finer particles (23), but this method is subject to the general errors of extrapolation,

and corrections for particle shape must be applied. Such extrapolation methods are only applicable to fairly homogeneous substances, and the heterogeneous nature of coal makes them unsuitable.

Sedimentation. Particles through 200 mesh may be measured by rates of sedimentation and calculations based on Stokes' Law. This requires a suitable medium, with attendant difficulties where coal is concerned, for as mentioned previously, coal is not readily wet with water and use of organic liquids may result in solvent action.

In any case the sedimentation method is tedious and none too accurate, and is only satisfactory with particles of uniform size and shape.

Elutriation by either air or liquid suspension is subject to the same objections as sedimentation, and although the method is applicable to particles finer than can be measured by sedimentation, the difficulties when applied to coal are too great for satisfactory use.

Hydrometer Method. There have been recent developments in wet mechanical analysis, culminating in the "Hydrometer Method" of Buoyoucos (24). The determination is a direct hydrometer measurement of the density of a suspension of fine particles after successive time intervals of settling. Such analysis measures the separation of any granular material into several fractions of different grain size, or gives the indirect determination of the whole grain size distribution.

It might be noted that this procedure is used in the soils work of the Agriculture Department of this University, and a modification developed by Casagrande (25) is used by the Department of Civil Engineering.

These methods, though seemingly satisfactory for soils work, are not suitable for coal, due to the difficulty of wetting, and the uncertainty due to the buoyancy effect of the gas enclosed in the capillaries

of the coal.

Microscope Method. The method of measuring particle size accepted as being the most accurate is microscopic examination. The objections to this method are the time required and the tediousness of the determination coupled with the difficulty of securing a representative field for examination. As the field is extended the work increases to such an extent as to prohibit the use of the microscope for a practical determination of particle size.

The microscope is suitable for checking purposes, however.

An optical method (26) for determining the area of particles might be mentioned. A photocell is so arranged that shadows cast by the particles vary the amount of light that falls on the cell. It is reported as being suitable to sizes down to 8 microns in diameter, but entails the use of a rather complicated apparatus, and its applicability to coal dust is doubtful.

Bulk-density Method. When coal is ground in a ball mill its bulk-density visibly decreases. Sinnatt and Slater (27) used this fact in a method for determining the fineness of grinding of coal. They determined the weight of a standard volume of coal dust to give a "fineness figure" for each coal sample. This method, however, allowed for no comparison between different samples, and was at best only a comparative measure of fineness.

It would appear that none of the above mentioned methods is suitable for the determination of the particle size of finely ground coal.

A method has been developed during this investigation which gives a measurement of the specific surface of coal dust, which can be taken as a measure of the fineness of grinding. If the average particle size is

desired, it is necessary to assume a shape factor.

Determination of Specific Surface.

Development of the Method. The method is based upon an equation relating specific surface and permeability, deduced by Kozeny (28) from purely theoretical considerations. The original work of developing the method was done by Carman (29). He reviewed the more reliable data for the permeability of granular beds in the light of Kozeny's equation, and showed that experiment and theory were in extremely good agreement.

The "permeability" is defined as the linear rate of flow of liquid through a bed under unit hydraulic gradient, and by D'Arcy's law, the linear rate of flow through a porous medium is proportional to the pressure gradient across the bed.

$$\text{Expressed as an equation } K = \frac{Q}{A} \cdot \frac{L}{h}$$

where: K = permeability
Q = quantity of liquid flowing through the bed in unit time.
A = area of cross section of the bed.
L = depth of the bed.
h = pressure drop through the bed.

Carman relates permeability and specific surface in a serviceable form of Kozeny's equation as follows:

$$So = 14 \sqrt{\frac{1}{KV} \cdot \frac{e^3}{(1-e)^2}}$$

where: So = specific surface = surface area per unit volume of actual material comprising the bed.
K = permeability as before.
V = kinematic viscosity of the liquid = absolute viscosity ÷ density.
e = porosity = free volume in the bed expressed as a fraction of the total volume.

It should be noted that $So = S + (1-e)$ where S = surface area per unit volume of the bed.

In a second paper (30) Carman describes a thorough experimental

investigation of the applicability of Kozeny's equation to granular materials. He tested the effect of varying particle shape, viscosity, porosity, head of liquid and depth of bed. As mediums he used such liquids as water, acetone, benzene, carbon tetrachloride, ethyl alcohol and ether. He also made tests with air.

He found one limiting factor, - the equation holds only for conditions of streamline flow, the criterion being that $Q + AVS$ is less than 2. However, for all ordinary experimental conditions he found the equation valid.

Carman also tested the accuracy of the experimental results by using materials of measured specific surface, and found that the error did not exceed 3 - 4%. This is probably far in advance of the accuracy of the systems cited for measuring particle size of irregular particles.

In this second paper Carman describes work with a minimum size of 0.1 mm. diameter, but in a third paper (31) he describes tests down to an average particle size of 2-3 μ . He found that the method was accurate to plus or minus 5%, regardless of the shape and size distribution of the particles.

Carman's conclusions are verified in general by the work of Wiggins, Campbell and Maass (32) who measured the specific surface of fibrous materials by this method.

CURRENT INVESTIGATION

An investigation was carried out in these laboratories, with Carman's work as a basis, to determine if the permeability method could be used to measure the specific surface of coal dust. In his later work Carman mentioned having used air in place of the usual liquids, but gave no details. However, in view of the difficulty of using liquids

with coal, it was decided to try to adapt Carman's method to the use of air.

Apparatus and Procedure.

The apparatus used is shown in figures XIV, XV and XVI. The permeability cylinder first used was number 1 in figure XVI, but later this was replaced by number 2.

The following procedure is applicable to both.

A weighed quantity of dust is placed in the permeability cylinder (fig. XV) A, and packed and levelled by tapping, or later by the use of a plunger. Air is drawn through the bed of dust in A at a constant rate, controlled by a constant head, which can be varied by adjustment of F. The manometer B measures the pressure drop through the bed, while the manometer E measures the pressure inside the bottle C. The fritted glass bubbler D gives very fine air bubbles, and thus prevents the pulsations of the liquid in the manometer B. The thermometer H measures the temperature of the system.

The time taken to collect a given volume of water in the volumetric flask G is measured by a stopwatch. For most of the determinations a burette was used in place of the volumetric flask.

When samples of dust which had been previously dried were tested, dry air was drawn through the cylinder, as moisture adsorbed by the coal might change the permeability of the bed.

For dusts finer than 200 mesh, a suitable thickness of bed was 2 - 4 cm., but for coarser particles a greater depth of bed was necessary for accurate results.

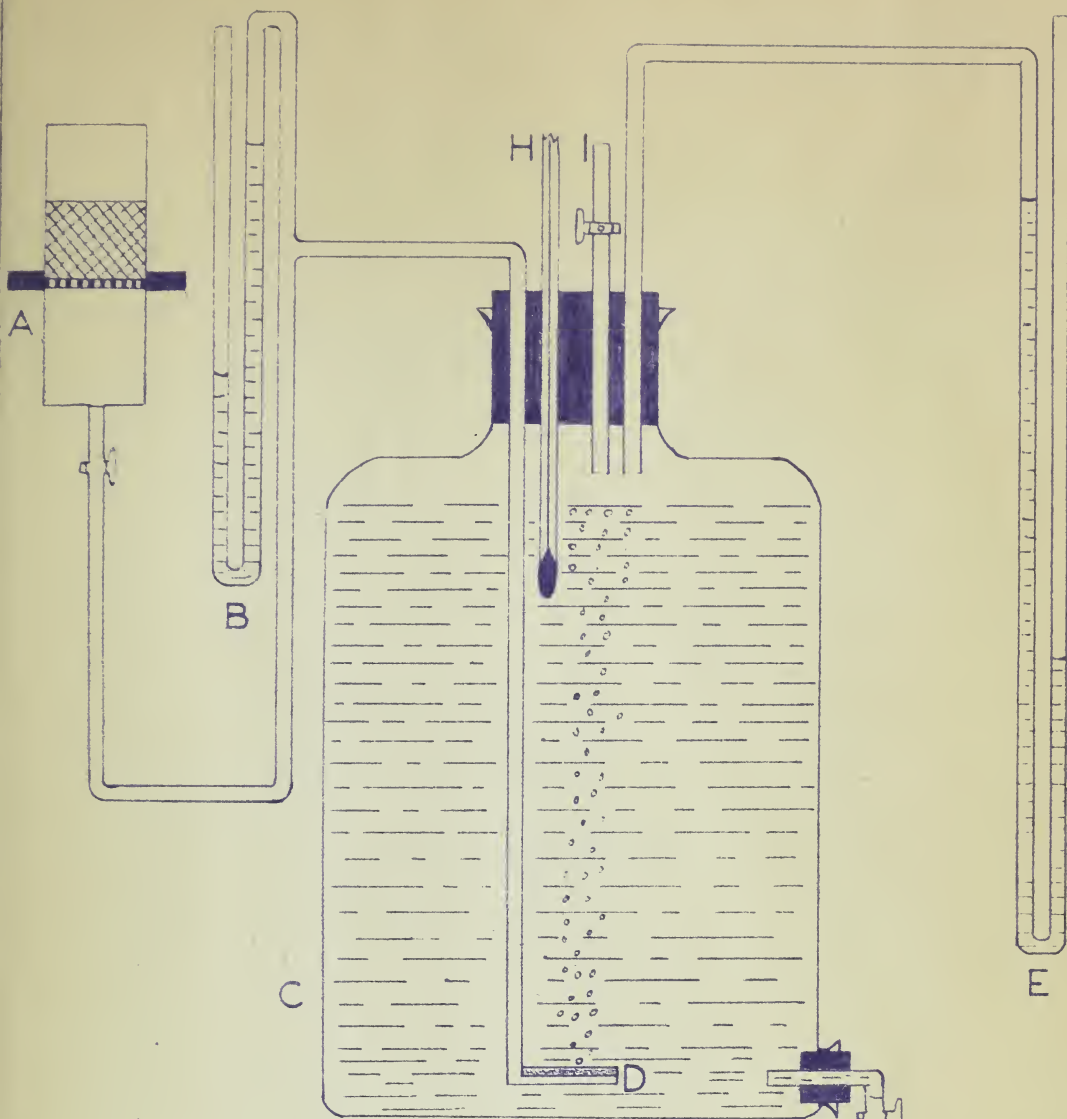
Method of Calculation.

The volume of air drawn through the bed of dust is calculated from the volume of water collected in G, with allowances for the air pressure



Figure XIV. Photograph of Permeability Apparatus

FIGURE XV
Diagram of Permeability Apparatus



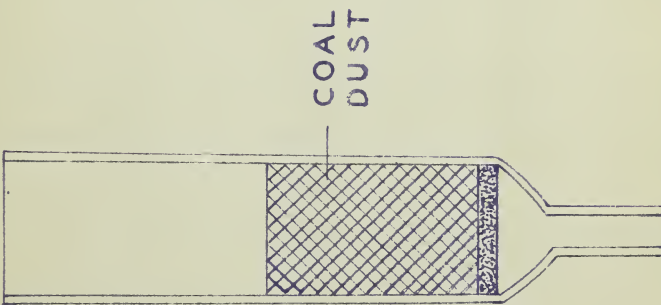
- A- PERMEABILITY CYLINDER
- B- MANOMETER (WATER)
- C- CONSTANT SUCTION BOTTLE-10L.
- D- FRITTED GLASS BUBBLER
- E- MANOMETER (WATER)
- F- SUCTION CONTROL OVERFLOW
- G- VOLUMETRIC FLASK - 250ML.
- H- THERMOMETER
- I- PRESSURE RELEASE TUBE

FIGURE XVI

Permeability Cylinders

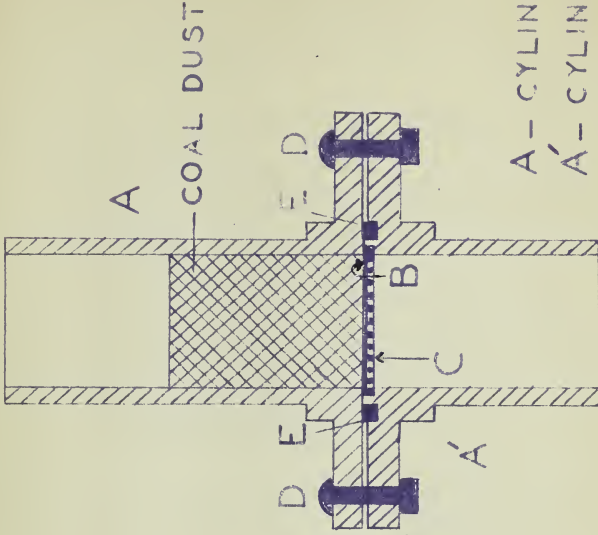
1. Fritted glass filter tube
2. Metal cylinder

1

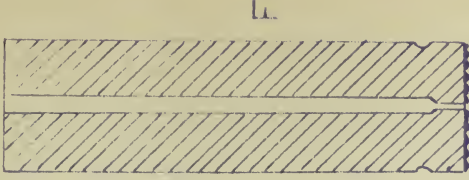


FRITTED GLASS
FILTER TUBE

2



- A - CYLINDER
- A' - CYLINDER BASE
- B - FILTER PAPER DISK
- C - PERFORATED BRASS DISK
- D - BOLTS
- E - RUBBER GASKET
- F - PLUNGER



and the water vapor pressure in C. This volume is calculated to the mean pressure of the bed, and, divided by the time recorded by the stopwatch, gives the "Q" of D'Arcy's equation. The area of cross-section "A" and the depth of "L" of the bed are measured, and "h", the pressure drop across the bed, is expressed in terms of the fluid flowing through the bed. Thus the permeability "K" is calculated according to the equation

$$K = \frac{Q}{A} \cdot \frac{L}{h}$$

where: Q is expressed in c.c. per second
A is expressed as sq.cm.
L is given as cm., and
h as cm. of water pressure divided by the density of air
(0.00108) in the bed.
g/ml.

The porosity "e" is then calculated from the weight and specific gravity of the dust and the volume of the bed, as

$$1 - \left(\frac{\text{volume of coal}}{\text{volume of bed}} \right)$$

and from this the porosity factor $\frac{e^3}{(1-e)^2}$ may be calculated.

The specific surface So is then calculated by means of the original equation

$$So = 14 \sqrt{\frac{1}{KV} \cdot \frac{e^3}{(1-e)^2}}$$

Throughout the experiments two values were taken as constants:
density of air = 0.00108 g. per ^{c.c.} ~~litre~~, and kinematic viscosity of air = 0.167 stokes, since the possible variations in these values caused by changes of temperature pressure and humidity in the laboratory were well within the experimental error.

The specific gravity of the dust was calculated by the method described in Section A.

The average particle size can only be calculated from the specific surface by assumptions as to sphericity. If the particles are spheres,

the average diameter would be equal to 6 ± 50 cm., but for particles of irregular shape a "sphericity" factor must be applied.

Gaudin (33) defines the degree of sphericity of a particle as the ratio of the surface of a sphere which has the same volume as the particle to the actual surface of the particle. He lists the sphericities of various naturally occurring particles of definite shapes, which range from 1.00 for a sphere to 0.22 for flat disks of mica, and suggests that for particles encountered in mineral dressing problems the sphericities would be of the order of 0.5 to 0.7.

It was found difficult to determine the sphericity of coal dust even with microscopic examination, but in the writer's opinion the sphericity is probably within the above limits of 0.5 to 0.7.

Determinations of Specific Surface

The first determinations of specific surface were made using the fritted glass filter tube (fig. XVI) as the permeability cylinder. It was soon found that a variable correction had to be made for the resistance to air flow of the fritted glass.

Accordingly a series of runs was made with the empty cylinder to determine the pressure head required for various rates of flow. The pressure head was then plotted against the linear rate of flow to give a correction curve for the fritted glass.

The values determined are listed in table XI and the correction curve shown in figure XVII.

As shown by figure XVII the correction "curve" is actually a straight line, indicating the accuracy of the measurements of both pressure head and rate of flow.

For all subsequent determinations with the fritted glass tube, the

corrections to be applied were taken from the curve.

TABLE XI
Correction Data for Fritted Glass Disk

Pressure head (cm. of water)	Linear rate of air flow (cm. per sec.)
0.13	0.0026
0.40	0.0076
0.80	0.0146
1.35	0.0243
2.07	0.0364
2.64	0.0468

Specific surface determinations were first made on samples of sand previously sized by screening.

For each sample of material a number of runs was made to check the constancy of results with variations of conditions. Thus with one weight of sample and porosity of bed the rate of flow would be measured for two or three different pressure heads to check the permeability. Then a different weight of sample with, in general, a slightly different porosity would be used, with variations in pressure head as before.

Results of these determinations are given in table XII.

Determinations were also made on a sample of Ottawa sand, a sample of ore dust, and a sample of Brazeau coal dust sized to 150-200 mesh. Results for these determinations are also shown in table XII.

The determinations of specific surface were in fair agreement, but the deviations were too great for satisfactory results. It was thought that these deviations were probably due to errors in measuring the dimensions of the bed.

The large specific surface of the Brazeau coal dust was thought to indicate a very low sphericity. However, this was partially explained later by microscopic examinations which showed that the sample contained

FIGURE XVII

Correction Curve for Permeability Cylinder #1

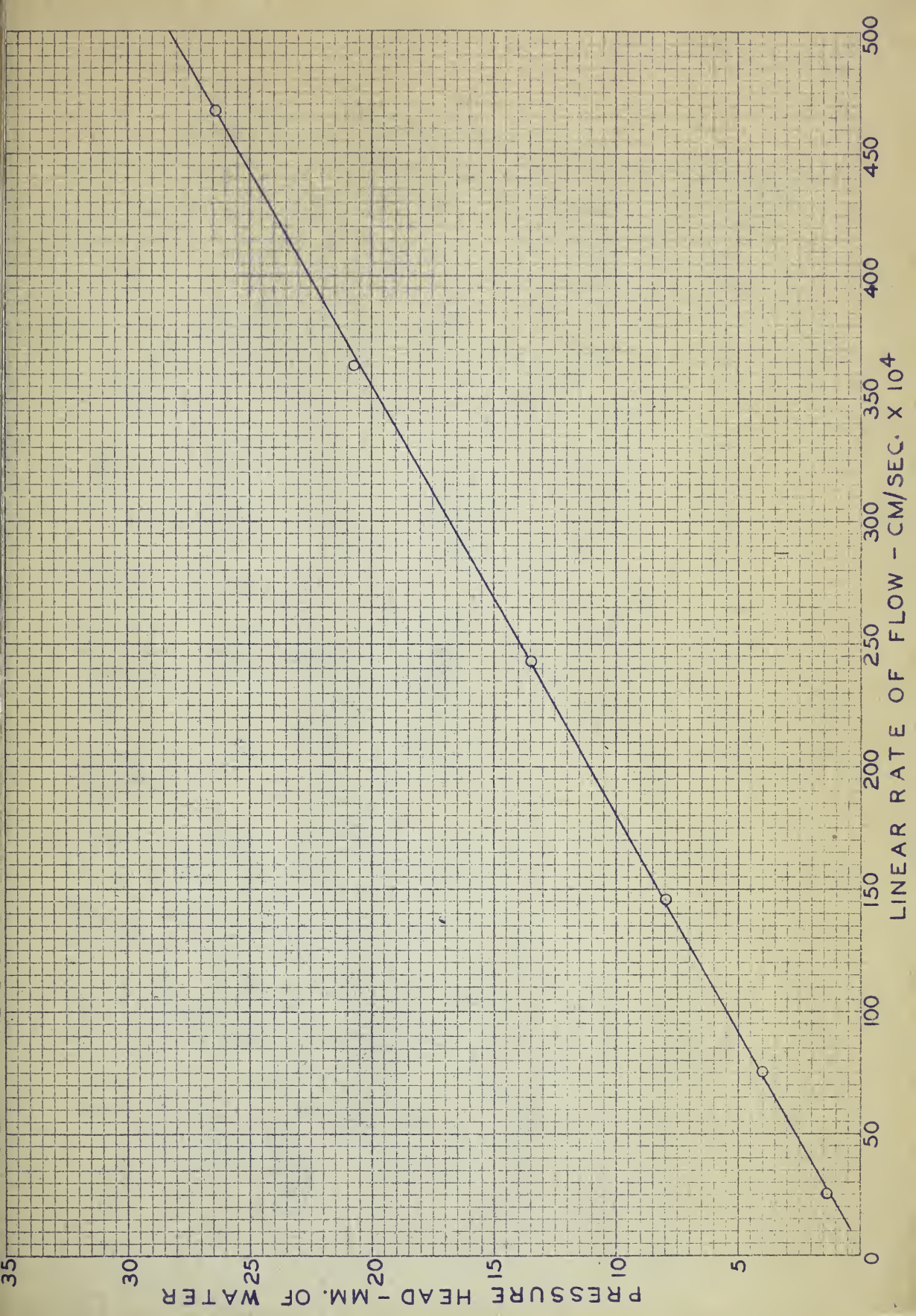


TABLE XII
Specific Surface Determinations.

Material	Wt. of sample (grams)	Porosity	Pressure head (cm. of water)	Permeability $K \times 10^4$	Specific Surface S_s (sq. cm.)
Sand	16.189	0.390	0.27	6.04	556
100-150	16.189	0.390	0.39	7.56	498
mesh	29.059	0.419	0.63	8.38	552
	29.059	0.419	0.23	9.66	518
	29.059	0.390	0.71	6.37	542
	29.059	0.390	0.27	5.49	583
	29.059	0.390	0.49	6.39	541
Average					541
Average deviation %					3.5 %
Sand	19.342	0.458	0.44	5.17	862
150-200	19.342	0.458	0.33	5.11	868
mesh	29.314	0.456	0.70	6.24	775
	29.314	0.456	0.45	6.35	768
	29.314	0.422	0.91	4.34	780
	29.314	0.422	0.50	4.11	810
Average					808
Average deviation %					4 %
Brazeau	8.552	0.466	0.13	1.88	1490
coal dust	8.552	0.466	0.87	1.93	1470
150-200	8.552	0.466	0.38	1.88	1490
mesh	8.552	0.423	0.51	0.88	1670
	8.552	0.423	0.27	0.97	1650
	8.552	0.423	0.99	1.10	1550
Average					1550
Average deviation %					4.5 %
Ottawa	9.987	0.425	4.17	0.096	5320
sand	9.987	0.425	2.88	0.095	5340
minus 200	16.798	0.401	6.16	0.094	5540
mesh	5.761	0.439	3.48	0.086	6070
	5.761	0.439	1.88	0.088	5990
Average					5650
Average deviation %					5.3 %
Ore dust	7.233	0.573	3.21	0.091	11500
minus 200	7.233	0.573	1.85	0.093	11400
mesh	13.138	0.522	5.55	0.065	10650
	13.138	0.522	2.16	0.065	10600
Average					11040
Average deviation %					3.5 %

a fair percentage of particles of minus 200 mesh size.

At this time some recent work of Lea and Nurse (34) was first noted. They also had adapted Carman's method to use air. Their permeability cylinder seemed better suited to this work than the fritted glass tube, and with a few minor changes was adopted as shown in figure XVI, cylinder 2.

The bed of dust in this cylinder is supported by a filter paper on a perforated disk. The dust is packed uniformly by rotating the plunger, and the depth of bed is measured accurately by a vernier scale that can be attached to the plunger.

It was found that for measurements of permeability of fine dust no correction was required for the resistance of the filter paper to any air flow used.

Details of a sample determination follow.

Sample: Edmonton coal dust, air dried, ground 70 hours
in a special ball mill.

Specific gravity of dust = 1.47

L = depth of bed = 3.13 cm.

A = area of cross section = 3.77 sq.cm.

Weight of dust in bed = 7.001 g.

Volume of bed = $3.13 \times 3.77 = 11.80$ c.c.

Volume of coal = $7.001 \div 1.47 = 4.76$ c.c.

$e = \text{porosity} = 1 - \frac{4.76}{11.80} = 0.596$

$\frac{e^3}{(1-e)^2} = \text{porosity factor} = 1.30$

Volume of water collected = 36.25 ml.

Time = 720 sec.

Vapor pressure in suction bottle = 15 mm. of mercury

Pressure in suction bottle = 40 mm. of mercury below atmospheric
Barometer reading = 706 mm. of mercury

Pressure drop through bed = 15.10 cm. of water = 11.1 mm. of mercury
Mean absolute pressure in bed = $706 - 5.6 = 700$ mm. of mercury

$$\frac{Q}{A} = \text{linear rate of air flow} = \frac{36.25}{3.77} \times \frac{651}{700} \times \frac{1}{720} = 0.0124 \text{ c.c. per sec.}$$

$$V = \text{kinematic viscosity} = 0.167 \text{ stokes}$$

$$h = \text{pressure drop through bed (expressed in terms of air of average density } 0.00108) = \frac{15.10}{0.00108} = 13980 \text{ cm.}$$

$$K = \text{permeability} = \frac{Q}{A} \cdot \frac{L}{h} = 0.0124 \times \frac{3.13}{13980} = 2.77 \times 10^{-6} \text{ cm. per sec.}$$

$$So = \text{specific surface} = 14 \sqrt{\frac{1}{KV} \cdot \frac{e^3}{(1-e)^2}} = 14 \sqrt{\frac{1.30 \times 10^6}{0.167 \cdot 2.77}} = 23,500 \text{ sq.cm.}$$

$$\text{Average diameter, assuming a sphericity of } 0.6 = \frac{6}{So} \div 0.6 = \frac{6}{23,500} \times \frac{1}{0.6} = 4.25 \times 10^{-4} \text{ cm. or } 4.25 \text{ microns}$$

Four separate runs, with varied conditions, were made in this apparatus with each dust tested. These four runs could be completed in two hours or less, depending on the rate of air flow advisable. The tests could be made on from 5 to 20 grams of material, an amount far less than that required for screen analysis.

Repeat determinations were made on ordinary sand, Ottawa sand and Brazeau coal dust with results shown in table XIII. Determinations were also done on Edmonton coal dust screened to 150-200 mesh, and on the very finely ground coal dusts described in Section C, with representative results also shown in table XIII.

The results are described later, but they show in brief, smaller deviations than with the earlier apparatus, and no increase in deviations in tests with very fine dust.

Examination by Microscope.

Some microscopic examinations were carried out on various dusts to obtain some comparison with the specific surface measurements. Magnification of 1200 was used, with an oil immersion objective. The scale of the micrometer eyepiece was calibrated with a micron gauge to permit

TABLE XIII
Specific Surface Determinations Using New Cylinder

Material	Wt. of sample (grams)	Porosity	Pressure head (cm. of water)	Permeability $K \times 10^6$	Specific Surface So. (sq.cm.)
Sand	20.570	0.449	1.40	773	670
100-150 mesh	20.570	0.449	0.93	777	669
	20.570	0.449	0.50	744	684
	22.300	0.459	0.91	804	694
Average					679
Average deviation %					1.5 %
Edmonton coal	9.347	0.519	2.90	375	1,380
dust	9.347	0.519	1.12	344	1,440
150-200 mesh	9.661	0.525	2.34	396	1,370
	9.661	0.525	1.03	405	1,360
Average					1,390
Average deviation %					2 %
Sand	15.388	0.467	0.32	499	917
150-200 mesh	15.388	0.467	1.10	468	947
	15.388	0.467	1.42	565	862
	21.355	0.464	3.65	469	934
	21.355	0.464	2.30	475	927
	21.355	0.464	1.10	403	1,006
	21.355	0.464	2.54	485	919
Average					930
Average deviation %					3 %
Brazeau coal	8.765	0.486	3.92	141	1,900
dust	8.765	0.486	2.40	152	1,830
150-200 mesh	8.765	0.486	5.50	147	1,860
	10.108	0.489	6.35	148	1,890
	10.108	0.489	4.02	151	1,870
	10.108	0.489	1.61	153	1,850
Average					1,870
Average deviation %					1 %
Ottawa sand	20.325	0.470	12.53	11.1	6,260
minus 200	20.325	0.470	6.94	11.6	6,120
mesh	20.325	0.470	4.10	11.5	6,140
	20.325	0.470	12.50	11.3	6,180
Average					6,180
Average deviation %					1 %
Edmonton coal	7.114	0.599	16.13	3.94	20,000
dust	7.114	0.599	13.08	3.98	19,900
(ground 30	8.611	0.585	12.61	3.37	20,200
hrs.)	"	"	16.32	3.36	20,100
Average					20,050
Average deviation %					0.5 %

Table XIII - continued

Material	Wt. of sample (grams)	Porosity	Pressure head (cm. of water)	Permeability $K \times 10^6$	Specific Surface S_o (sq. cm.)
Edmonton coal	7.001	0.596	15.10	2.77	23,500
dust (ground	7.001	0.596	10.30	3.13	22,000
70 hrs.)	9.077	0.593	17.90	2.78	23,100
	9.077	0.593	15.94	2.93	22,500
Average					22,800
Average deviation %					2 %
Edmonton coal	6.269	0.570	11.80	1.35	29,400
dust (special	6.269	0.570	16.52	1.37	29,300
dry grind)	7.793	0.558	16.57	1.19	29,500
	7.793	0.558	11.66	1.22	29,100
Average					29,300
Average deviation %					0.5 %

direct size readings; one scale division represented approximately 2 microns.

No satisfactory comparison could be made, due to the difficulty in securing a representative field. The larger particles, such as the 150-200 mesh sand, were dusted directly onto the slide. With coal dust, the best results were obtained by forming a dilute suspension of the dust in alcohol, shaking, and quickly transferring a drop of the suspension onto a slide. The alcohol on drying left a fairly representative field of particles for examination.

It was found that the coal dust supposedly sized by screening to 150-200 mesh still contained a fair percentage of minus 200 mesh particles, thus at least partially accounting for the abnormally high specific surface measurements of the permeability tests. It was also noted that in the ordinary minus 200 mesh dusts, particle sizes ranged approximately from 1 to 100 microns. However, the percentages of particle sizes varied from sample to sample, as was indicated by the specific surface measurements. In the most finely ground dust obtained, a few particles of 10-20

microns were still observed, although most of them were less than 5 microns.

This is illustrated in the photographs of figure XVIII. Photograph 1 is a picture of the field of a dust which had a specific surface of 11,400 sq.cm. (or a particle size of 8.5 microns diameter, if the sphericity = 0.6), while the dust of photograph 2 had a specific surface of 21,300 sq.cm. (and a particle size of 5 microns diameter, if the sphericity = 0.6). Both photographs show a range of sizes, but the percentage of small particles is much greater in the second dust that had nearly twice the specific surface.

It might be noted that the coal dust particles gave an appearance of fairly regular shape quite similar to that of sand.

A comparison of approximate sizing is given in table XIV.

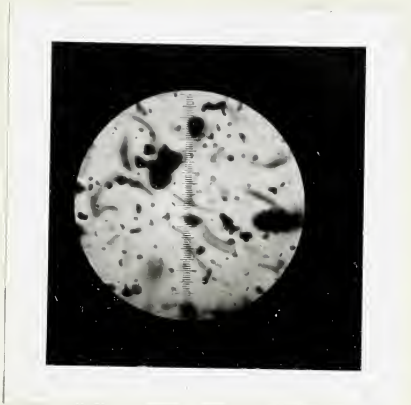
TABLE XIV
Comparison of Particle Size Measurements (diam. in microns)

Material	Screen size range	Microscope size range	Determined size *
Sand	147 - 104	100 - 110	126
Sand	104 - 74	80 - 100	92
Ottawa Sand	Less than 74	10 - 20	14
Coal dust (ground 30 hrs.)	Less than 74	Up to 20 (Mostly 2 - 4)	5

* Sphericity of sand assumed to be 0.7
and of coal, 0.6

Sphericity.

The Tyler screens of 100 and 150 mesh have openings of 0.147 and 0.104 mm. The specific surface of spherical particles of the mean size of 0.125 mm. is 482 sq.cm. Similarly the value for 150 - 200 mesh screens is 674 sq.cm.



Photograph 1



Photograph 2

FIGURE XVIII

From this, the sphericity of the sand, given by the specific surface as determined is 0.71 and 0.72 for the two sizes respectively. For the Edmonton coal dust sized to 150-200 mesh the sphericity would be 0.485, while for the Brazeau dust of the same size it would be 0.361.

In the consideration of these sphericities, possible interference due to incomplete screening should not be forgotten, nor the fact that the mean diameter of particles of different screenings will not always approximate the mean between the limiting screen apertures. With particles of irregular size the smallest diameter may be the controlling factor in screening, so that it is possible for particles to pass through a screen opening smaller than the ^{average} diameter.

It might be noted that the numerical data indicates a sphericity of coal of less than 0.5, but in view of the above uncertainties, and the fact that microscopic examination suggests a regular shape tending toward cubical, a sphericity of 0.6 will be taken until further information is obtained.

The averaged values from table XIII, for some of the materials tested, have been collected in table XV with data to show the effect of different sphericity assumptions on the calculated mean diameter.

Fineness Factor Comparison.

Determinations of specific surface were made on 5 samples of coal mine dust to compare the results with the fineness factors computed from the screen analyses by the U.S. Bureau of Mines method (35).

The determined specific surfaces of these dusts were found to vary as much as from 1.3 to 1.8 times the numerical values of the corresponding calculated fineness factor. This is not surprising as the fineness factor does not take cognizance of variations in size of the minus 200 mesh

TABLE XV
Screen Size, Specific Surface, Sphericity and Particle Size

Screen	Average opening (microns)	Test Material	Sp.Surface det.sq.cm.	Average diameter (microns)* Sphericities			
				0.5	0.6	0.7	1.0
100-150 mesh (147-104 u)	125	Sand	679	176	147	126	88
150-200 mesh (104-74 u)	89	Sand	930	129	108	92	65
	89	Edmonton coal dust	1,390	87	72	62	43
	89	Brazeau coal dust	1,870	64	54	46	32
Minus 200 mesh (less than 74 u) "		Ottawa sand	6,180	19	16	14	10
		Ore dust	11,000	11	9	8	5.5
		Edmonton coal dust (30 hr. grind)	20,000	6	5	4	3

* The average diameter is calculated, assuming the sphericities shown.
material.

It is suggested that specific surface would be a better guide to relative inflammabilities of mine dusts than is the fineness factor. Moreover the former can be more quickly determined.

Discussion of Results.

The slightly higher measurements of specific surface given by the second permeability cylinder indicate some constant error in one or both, but the general agreement is good.

The concordance of sets of results throughout, even for the very fine dusts, indicates the high degree of relative accuracy obtainable with the second cylinder and this method.

The concordance of sets of results throughout, even for the very fine

dusts, indicates the high degree of relative accuracy obtainable with the second cylinder and this method.

The difficulties of proving the absolute accuracy of the method are great. However, comparisons with screen sizings and with microscopic examinations indicate at least a general agreement, while the measurements of sphericity for sand agree with accepted values.

Lea and Nurse (34) report agreement of check results by air permeability similar to those obtained in this investigation. They show also that other gases such as hydrogen, nitrogen, or carbon dioxide are suitable. They give a comparison of specific surface measurements made by the air permeability method and by a sedimentation method, and show good agreement down to 5 microns. Below that they suggest that the sedimentation method is not reliable.

Conclusions.

This method appears to afford a convenient and simple means for measuring surface area of dusts, a measurement of considerable importance in many fields, although it does not give a particle size distribution.

The development of this method thus opens up a whole new field of investigation, especially with respect to the correlation of the reactions of finely ground coal with its surface area.

S E C T I O N C
FINE GRINDING OF COAL

Introduction.

For routine laboratory testing of coal a sample must merely be crushed to pass a 250 micron sieve. This is usually done by grinding in an Abbe' ball mill. However, for such work as investigations of oxidation, extraction, and solubility, Fischer (17) suggested^{use of} a special type of vacuum ball mill, indicating that in an ordinary ball mill, coal can only be ground to a certain fineness.

On the basis of this "ultimate fineness" an investigation of oxidation of coal had been started in the laboratories of the Research Council of Alberta, assuming a coal ground to this "ultimate fineness" as a standard with which to work. Results indicated, however, that this final point was never reached, so further investigation was postponed until a method of evaluating the fineness of grinding could be developed.

In preparation for further investigation a ball mill patterned after Fischer's μ mill was designed and constructed, and was put into use when the current investigation of μ coal was started.

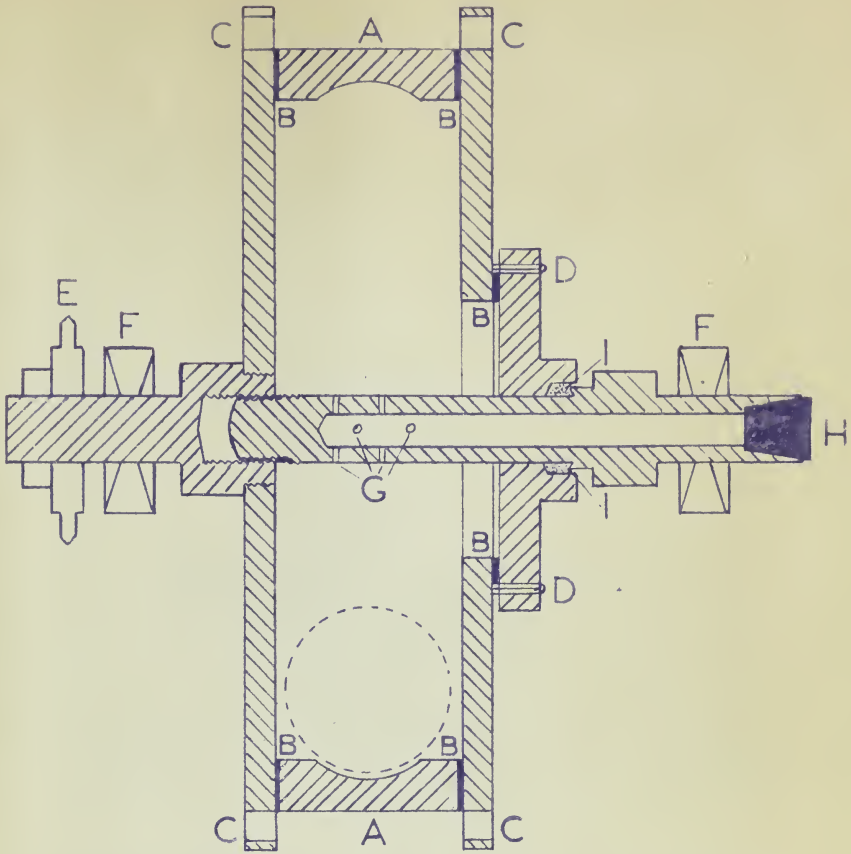
μ Ball Mill.

This was of a type of a single row of balls running in a groove as shown in figure XIX. A photograph of the assembled mill is shown in figure XX. The mill was made of a standard commercial ball race, and the hard steel balls employed were those supplied for this ball race. The ball race was clamped between two steel plates held together by 12 bolts. A hand hole in one plate was closed with a cover plate centered by 4 pins. One end of the axle was attached to one steel plate, while the other end passed through the cover plate and was used as a bolt to

FIGURE XIX

Cross sectional diagram of the μ ball mill, Fischer pattern,
designed by E. Stansfield.

Scale - half full size.



- A—OUTER RACE OF BALL BEARING
(SINGLE ROW DEEP GROOVE)
- B—GASKETS
- C—BOLT HOLES
- D—PINS FOR COVER PLATE
- E—SPROCKET WHEEL
- F—BALL BEARINGS
- G—HOLES FOR EVACUATION
- H—STOPPER
- I—PACKING



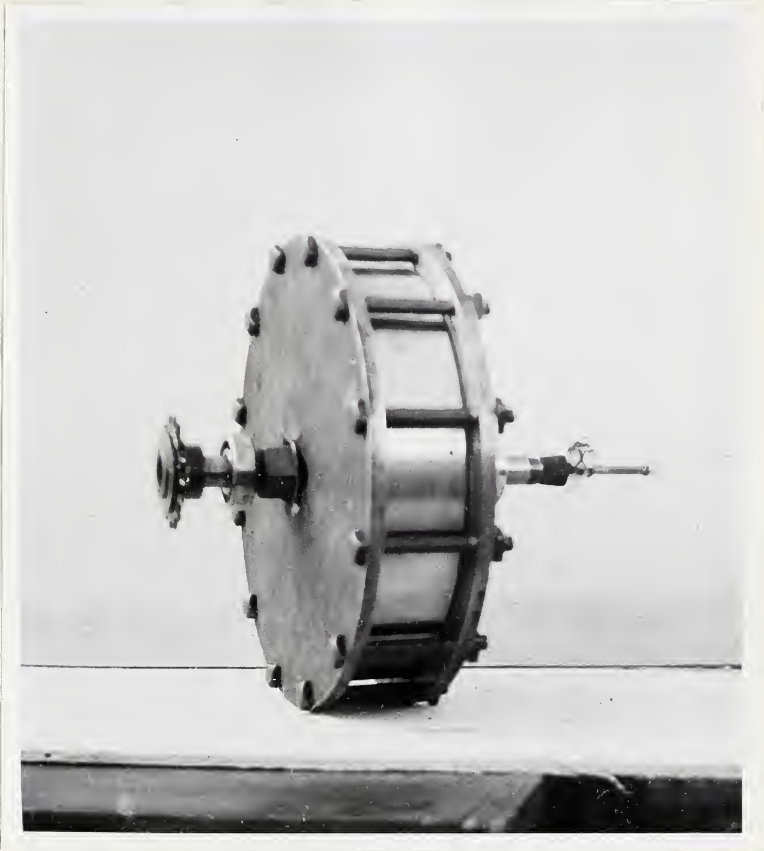


FIGURE XX

Photograph of assembled μ mill.



tighten the latter in position. One end of the axle was hollow to allow for evacuation of the mill when desired. The mill was driven by a chain and sprocket wheel and was provided with ball bearings. Gaskets and packing were provided as required for air tightness. The balls were $1 \frac{5}{8}$ inches in diameter and weighed about 285 grams.

By its design the mill grinds by rolling and crushing without the percussion effect of the ordinary Abbe mill. Not more than five balls, however, could be employed without tumbling under the conditions of use.

For grinding in vacuo a packing gland was arranged at H, connected by a metal tube to a mercury vapor pump backed by a vacuum oil pump.

The mill was suitably mounted, and was driven by an electric motor at the rate of 42 r.p.m.

Procedure.

The coal used for the grinding test was a sample from the Beverly mine, Edmonton, sized to 8-14 mesh. The coal had an ash content of 11.1% and was air dried to a moisture content of 7.5%.

A charge of 50 grams was used in each run, and runs^{made} of 10, 20, 30, 50 and 70 hours grinding in air.

Specific surface determinations were made on the product of each run, using the method described in Section B.

The results are given in table XV and shown graphically in figure XXI.

Grinding in Vacuo.

A series of grinding tests were then made with the ball mill evacuated. At first it appeared that the evacuation had some small beneficial effect on the grinding, but in tests with a semi-anthracite coal of low moisture content, no effect was observed.

FIGURE XXI

Grinding curve of an Edmonton coal, ground in air.

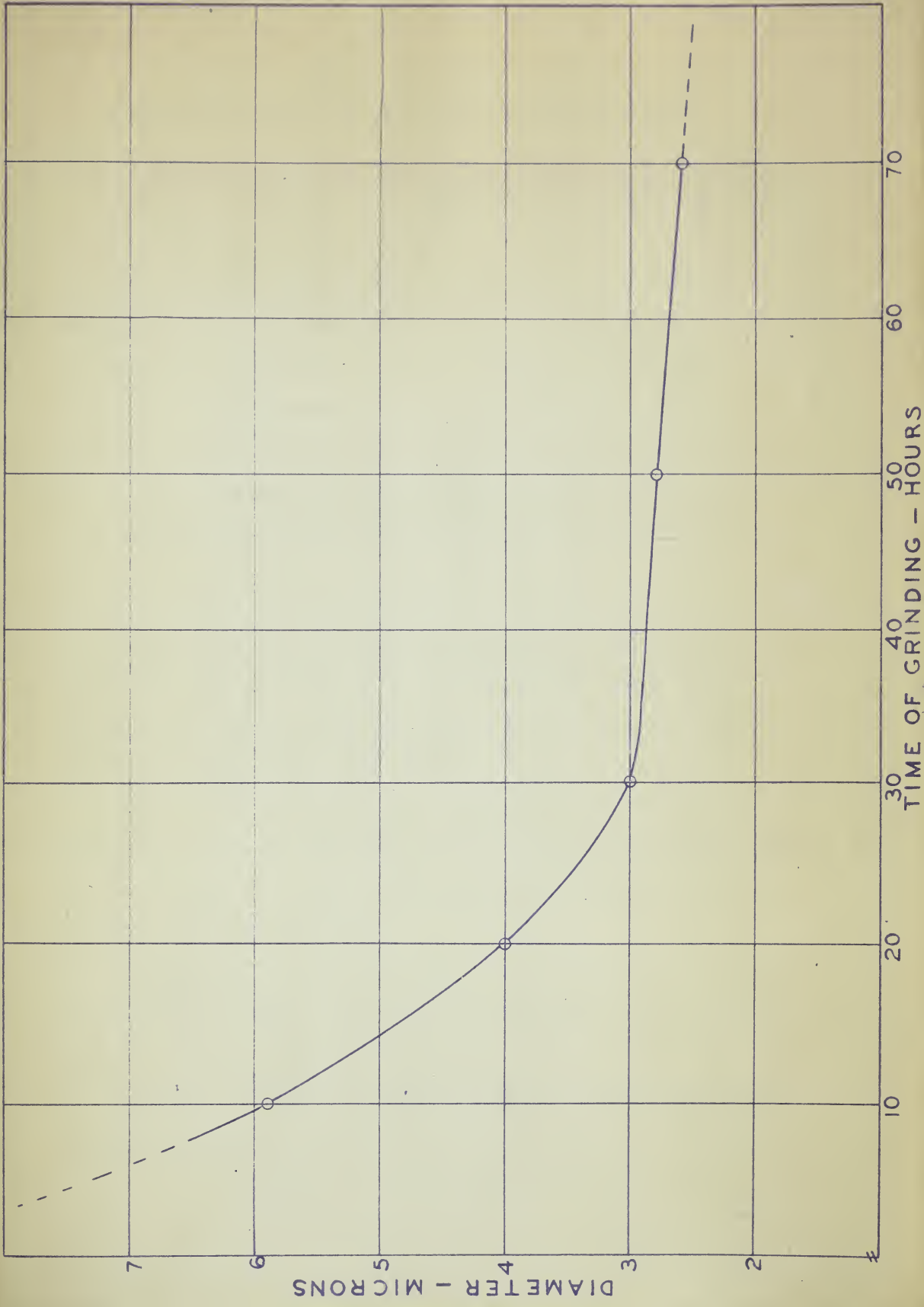


TABLE XV

Effect of Grinding on Specific Surface and Particle Size

Time of Grinding (hours)	Specific Surface So (sq.cm.)	Average Particle Size (microns) *	
		Sphericity = 0.6	Sphericity = 1.0
10	10,200	9.80	5.88
20	15,000	6.70	4.00
30	20,000	5.00	3.00
50	21,300	4.70	2.82
70	22,800	4.40	2.63

*Note: As investigations of sphericity had not been made at the time the grinding curve of figure XXI was plotted, the values shown are calculated on the basis of a sphericity = 1.0.

This indicated that possibly the evacuation was beneficial mainly because it removed moisture from the coal, and thus increased its grindability. Moisture determinations of portions taken at intervals during grinding supported this view.

A 250 g. sample of the Beverly coal was then thoroughly dried in a vacuum oven for 3 hours at 108°C. Then to prepare a homogeneous sample and to save time in later test grinding, the dry coal was pre-ground for 25 hours in an Abbe ball mill.

One 50 gram portion was then placed in the μ mill and ground for 10 hours in air, while a second 50 gram portion was ground in the same mill with a vacuum of only 0.06 mm. pressure.

Specific surface determinations were made on both products with results as follows.

	Specific Surface (Sq. cm.)	Average Particle Size (Sphericity = 0.6) (microns)
As pre-ground	25,800	3.90
Ground in air	29,000	3.45
Ground in vacuo	27,200	3.65

These results show no advantage in grinding in vacuo.

Grindability of Types of Coal.

Tests were then made to indicate the relative ease of grinding of types of coal. Four coals were selected for grinding - a semianthracite coal from the Cascade area, a medium volatile bituminous coal from the Mountain Park area, a high volatile C bituminous coal from the Lethbridge area, and a subbituminous C coal from the Edmonton area.

A 50 gram sample of each was ground for 50 hours in air in the μ mill, and specific surface determinations were made on the product. The results are shown in table XVI.

TABLE XVI

Grindability of Type Coals

Coal (area)	Ash content as ground %	Moisture content as ground %	Specific Surface (Sq. cm.)	Average Particle Size (microns) Sphericity = 0.6
Cascade	11.9	0.5	21,400	4.70
Mountain Park	9.4	1.0	27,300	3.65
Lethbridge	9.0	5.0	23,000	4.35
Edmonton	10.9	7.0	21,500	4.65

These results indicate no great difference in grindability of these types of coal.

It should be noted in relation to the moisture content that the effect of relative quantity of moisture varies with the difference in

classification. That is, ^{when ground,} a Cascade coal with 3% moisture would appear almost a paste, while an Edmonton coal with 3% moisture would appear dry.

As naturally occurring in the mines, a Cascade coal would contain less than 1% moisture, a Mountain Park coal about 1%, a Lethbridge coal about 10%, and an Edmonton coal about 20%.

Abrasion of Mills.

In investigations of finely ground coal it is essential that the grinding treatment should not contaminate the sample and thus change the analysis. In the regular work of the laboratory, where Abbe porcelain ball mills are used, with very hard "chromang" iron balls, no detectable increase of ash has been found. However, in some mills the reverse is the case.

A private communication from the Coal Research Laboratory of the Carnegie Institute of Technology at Pittsburgh said that when using a rubber lined pebble mill, with balls or rods of steel, to produce μ coal, there was a notable increase in ash. They ground the coal under water containing dilute alkali - presumably to overcome the difficulty of wetting. In a typical run, 750 grams of coal was ground, with 25,000 ml. of water containing 1.5 grams of caustic soda, for 72 hours at a speed of 50-60 r.p.m. The ash increased from 7.6 to 9.1%.

It is of interest to note that "no careful measurements on surface or exact size were made", but the product was examined microscopically.

In a test with the μ mill here, where 50 grams of coal were ground for 70 hours at 42 r.p.m., the determined ash percentages on a dry coal basis were 11.7 at the start, and 11.8 at the end. This mill has been operated for some 800 hours, and shows no visible wear of either race ^{of} balls.

Discussion of Results of Section C.

The curve of figure XXI indicates that Fischer's claim for an "ultimate fineness" of coal dust is doubtful and explains the findings in this laboratory during the oxidation work mentioned in the introduction to Section C.

The tests of vacuum grinding definitely show no effect on fineness of grinding from evacuation per se, but it was found that moisture content has a notable effect and evacuation of the mill does dry coals containing moisture.

From the result of the 25 hours pre-grind of the dry Edmonton coal in the Abbé mill, it would appear that an ordinary ball mill will give as fine a product as the μ mill.

For work on oxidation, study of occluded gases in coal, and for other work where it is important that the coal should not be exposed to air before test, use of the μ mill will be essential. It is also important that this mill does not contaminate the coal during grinding.

General Conclusions of Chapter IV.

The Permeability method of determining specific surface appears to be a practical method for evaluating the fineness of grinding of coal, and provides a suitable measure to which various reactions and tests of μ coal can be related.

The investigations described have provided the necessary foundation for a future study of finely ground coal. It opens up a large field of work relating the surface area to oxidation, solubility, extraction and so on.

Grindability tests may now be measured accurately. A study of

occluded gases may be made, using a modification of the μ mill-evacuation set up. Colloidal coal might also be investigated.

The importance of finely ground coal is shown in many recent reports.

Fischer (17) greatly increased his extraction yield by using μ coal. Juettner and Howard (36) using μ coal for distillation in vacuo got larger yields than with coarse coal. Asbury (37) greatly increased the rate of solvent action by using μ coal, while Landan and Asbury (38) found that μ coal gave a greater ultimate yield than did coarse samples with solvent extraction. (None of these investigators gave measurements of the surface area or particle size of the coals they studied).

All of these reports indicate the importance of investigations of μ coal and its related problems.

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